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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute of Petroleum was held at 61 New Cavendish Street, London, W.1, on 6 May 1959, the Chair being taken by H. Hyams, Vice-President of the Institute.

The General Secretary read the minutes of the previous meeting, which were confirmed and signed as a correct record. He also announced the names of members elected since the previous meeting.

The Chairman introduced the authors of the papers, which were presented in summary by J. W. Jackson, Operations Manager, BP Exploration Co. Ltd.

SUBMARINE EXPLORATION FOR OIL *

A Symposium by members of BP Exploration Co. Ltd.

I. INTRODUCTION

By C. N. CROSTHWAITE

THE huge gusher a mile and a half from the eastern shore of Lake Maracaibo, which in December 1922 flowed wild at 100,000 b.d. and blew a column of oil 200 feet into the air, was an event which not only put Venezuela on the oilman's map, but also, by providing the stimulus to prove the pools extending below the lake bed, gave birth to the idea of underwater oil production. The following year saw the first wells drilled in the shallow water along the shore and in the lake itself. Nearly every well gave flush production and the additional stimulus of competitive offsetting kept the fast new rotary drilling rigs in constant operation. New techniques were developed for drilling in water and by 1929, through use of wooden and concrete piles, wells were being sunk in 10 fathoms of water. In the course of some thirty years' development, several thousand wells have been drilled in the lake, and today the oil resources under the lake are regarded among the richest in the world, as is evidenced by the price of £1000/acre paid for a block in the lake centre when new concessions were offered two years ago for competitive bidding.

Offshore exploration, as distinct from drilling in inland waters, has had a relatively short history, but in the twenty years since 1938, when the first offshore oil discovery was made on the Louisiana Continental Shelf, this young branch of the oil industry has had a more rapid and at the same time a more spasmodic growth than any other.

The first substantial discovery off the Louisiana coast was made in 1947. The bright future for offshore growth that this opened up was immediately clouded by a six-year legal fight between the Federal and State Governments over ownership of state lands. With the passing by Congress in 1953 of the Outer Continental Shelf Act, whereby the rights of the States to the submerged lands within their traditional borders were recognized, drilling on a really large scale began in the Gulf of Mexico. From only thirty drilling units active in 1954 along the Texas and Louisiana coasts, by mid-1957 there were over 100 deep-water units—including more than thirty mobile ones—in operation. The number of wells drilled increased from 258 in 1954 to 491 in 1956, and in 1957 reached a peak of 665 well completions (Fig 1). The 1957 record would

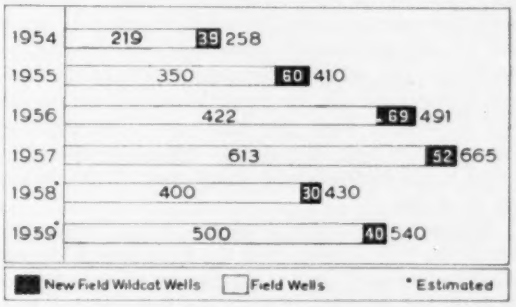

FIG 1

WELL COMPLETIONS

(Reproduced by courtesy of *World Oil*)

have been higher still had not the general economic situation brought about a sharp downturn in the last quarter of the year.

This downturn was not due to any disappointment with offshore results; indeed, the success ratio offshore has, as the figures quoted below will demonstrate, been its most attractive feature. In the latter half of 1957 the oil industry, in common with U.S. business in general, had to switch, if only temporarily, from expansion to drastic reduction of expenditure, and offshore ventures being substantially the more expensive, the cut was made where the payout is the longest way off.

Although operators have so far chosen their best prospects for drilling, the record of offshore successes in the Gulf of Mexico (Fig 2) is none the less very striking. Compared with the overall U.S. onshore ratio of

* MS received 10 February 1959.

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11 per cent, 39 per cent of the rank wildcats off Louisiana have produced oil or gas. 4 per cent (1 in 25) of offshore Louisiana wildcats found oilfields with crude

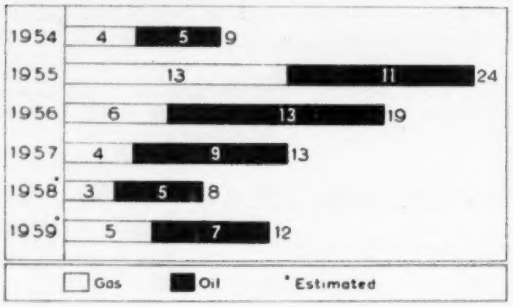

FIG 2

LOUISIANA OFFSHORE—NEW FIELD DISCOVERIES

(Reproduced by courtesy of *World Oil*)

reserves of 25 million brl or more each. This figure compares with a domestic average of about 1 in 500. Reserves proved up to the end of 1957 totalled 800 million brl of oil and 5000 billion cu. ft. of gas, an overall equivalent of 1200 million brl of oil. What the potential reserves in the Gulf of Mexico may be can as yet be only conjectural, since many of the offshore fields have had only the discovery well to prove them, and particularly has this been the case where the initial wells have been gas producers. The potential reserves have been estimated at around 10,000 million brl of liquid hydrocarbons and at least 50,000 billion cu. ft. of gas.

Although production from the entire Continental Shelf (the bulk coming from the Louisiana fields) has steadily increased until it reached an all-time high of 145,000 b.d. in 1957 (Fig 3), the industry has been

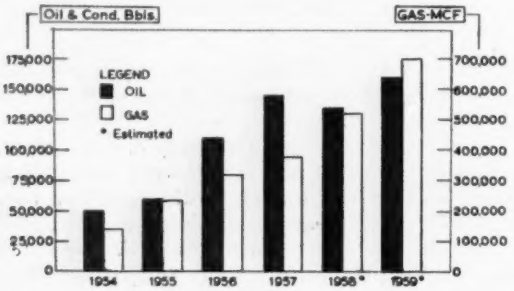

FIG 3

GULF OF MEXICO—OIL AND GAS PRODUCTION

(Reproduced by courtesy of *World Oil*)

ploughing back about four times as many dollars as it has taken out. The investment in capital and operating expenditures of all the companies, numbering more than seventy-five, engaged in offshore operations has totalled an estimated £714 million. Up to the beginning of 1958, combined income of oil and gas production has been less than £180 million. This very unfavourable ratio between expenditure and income has been due partly to increasing costs but more especially to sharply declining allowables. Well allowables have shrunk by approximately 68 per cent from those of 1953, *e.g.* the allowable production of a 9000–10,000-ft well is 162 b.d. as compared with 510 b.d. in 1953 (Fig 4). Because of the great expense of operating in deep water these adverse conditions have had a much more serious effect on offshore than on land operations. The present production allowed each well is clearly insufficient to yield a profitable return on the large investment necessary to drill the

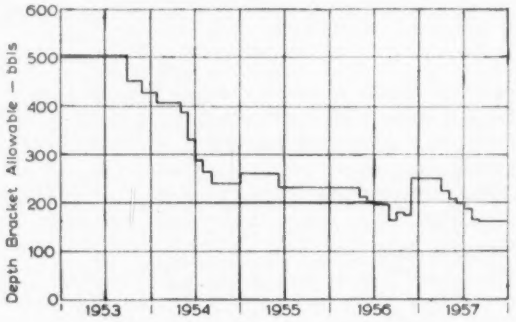

FIG 4

LOUISIANA OFFSHORE ALLOWABLES (WELLS 9000–10,000 FT)

(Reproduced by courtesy of *World Oil*)

well itself, let alone provide enough profit to build pipelines or carry on additional exploratory work. Industry leaders are in these circumstances appealing to the authorities to give offshore wells preferential allowables and permit wider spacing of wells; in other words, to permit offshore operators to produce as efficiently and economically as they know how. At the same time operators, by studying technological improvements and all other means of reducing expenditure, are facing the challenge of the cost problem.

There can be little doubt that as soon as the industry as a whole has resumed its forward march, exploration in the Louisiana offshore, regarded by many experts as the most promising area in the Continental U.S.A. for the discovery of major oil and gas reserves, will regain its former momentum.

The search for new underwater resources has been taking place in several other parts of the world.

Off the coast of Borneo, British and Dutch interests started in 1954 a drilling programme from fixed platforms at distances ranging up to 20 miles from the coast. Results so far have been disappointing, and drilling is temporarily suspended pending the arrival within the next few months of a new mobile platform.

It was also in 1954 that marine drilling, as distinct from deviated onshore wells, started in Trinidad. In 1955 the first Soldado offshore discovery was made by

T.N.A. in the Gulf of Paria, 14 miles off Point Fortin. So far, 14 producing wells have been completed, and they are now producing at a combined rate of somewhat in excess of 10,000 b.d. Offshore concessions now cover nearly all Trinidad's share of the Continental Shelf area. A subsidiary of the Standard Oil Co. of California holds licences covering much of the NW. part of the Gulf: after spending $1 million on geological and geophysical exploration of its offshore acreage this company is now carrying out a programme of widely spaced test wells.

The Soldado discovery stimulated intense competition for the new concessions offered two years ago on the Venezuelan side of the International boundary. A bonus of £36 million was paid by the Group of five U.S. companies which won the acreage (totalling 152,000 acres) nearest the Soldado discovery. It is relevant to mention here that 219,000 of the total of 380,000 acres in the Gulf of Paria offered by the Venezuelan Government were leased, and it may be inferred from this that many companies considered that such a premium on top of the high cost of offshore operations was too high a price in relation to their assessment of the value of the territory. Although time will show whether such a high price was justified, the "Paria Operations" Group got off to a promising start, having made a commercial discovery with their first well.

Turning to the Eastern Hemisphere, the waters of the Persian Gulf have over the past year been the scene of keen international rivalry. The high potentialities of this region, flanked by the great oilfields of Kuwait, Persia, and Saudi Arabia, need no emphasis.

The first Persian Gulf offshore discovery was Aramco's Safaniya field. Found in 1951, it can now produce at a rate of 175,000 b.d. 30 miles to the SE and some 9 miles offshore Aramco made a further discovery last year at Manifa, but this is still being developed, and further wells are being drilled there. In the southern part of the Gulf, 20 miles E of the island of Das and 80 miles N of the Trucial Coast mainland, Abu Dhabi Marine Areas Ltd (now generally known as ADMA, in which BP holds a two-thirds interest and CFP one-third, BP being the managing operators) has just completed its first well successfully as an oil producer and is now drilling further appraisal wells on the structure. These successes will have further enhanced the value of the offshore areas on the other side of the Gulf which the Persian Government still has available to offer. It was in August 1957 that the Persian Government granted the first concession in its Continental Shelf. This award, made jointly to ENI, the Italian State oil entity, and NIOC, was significant in that Persia was thereby the first Middle Eastern state possessing its nationally-owned oil company to take advantage of economic circumstances to alter the accepted organizational pattern. Under this agreement NIOC contributes capital towards the oil operation and gains part of the revenue thereof, whereas previously all the capital required along with technical skill had been produced by the foreign producing agent. The second offshore award of an adjacent area was made in June 1958 to Pan American Petroleum Corporation, again in a 50/50 joint venture with NIOC, but the financial conditions of the partnership were for the foreign partner more exacting, and included a cash bonus of £9 million. Meanwhile, on the other side of the Gulf, no less intense competition had been proceeding for Kuwait's and Saudi Arabia's interests in the offshore area of the Neutral Zone. A Japanese company signed an agreement with the Saudi Government in December 1957, and a counterpart concession with the Ruler of Kuwait was concluded in June 1958. The terms, which included provisions for the Governments to receive 57 per cent of net income in Kuwait's case and 56 per cent in Saudi Arabia's, were the most onerous yet accepted in Middle East operations.

When work on these new concessions gets into its stride, the Persian Gulf promises to rival the Gulf of Mexico as the world's most active offshore area. Such is competition between companies, and indeed between the nations, to secure new reserves of oil for the future that neither the much higher costs of offshore exploration nor more exacting operating terms deter the search under the sea bed in regions where the chances of establishing substantial reserves look bright.

II. GEOLOGY

By S. Elder

Marine exploration for oil began in those parts of the world where it was known or could reasonably be assumed that the submerged rocks were in effect an offshore continuation of proved oilfields or oil provinces. The area of exploration has since been widened to include offshore extensions of sedimentary areas in which oil has not yet been found but where the prospects of doing so are considered to be good. These are still the only marine areas which under present conditions can justifiably be explored for oil on economic or technical grounds.

Such areas fall into two broad categories. First, the sediment-filled structural or erosional depressions on the continental land-masses, below epicontinental seas, which almost certainly are underlain by rocks similar to those of the confining land. Where the

latter are proved or prospective sources of oil and the sea is shallow, then the prospects of the submerged rocks are high. Examples are Lake Maracaibo in Venezuela, the Persian Gulf, and the Caspian Sea. In these the relationship between sea and underlying and surrounding rocks has a long and complex geological history. On the other hand, epicontinental seas, such as Hudson Bay and the Baltic, which have had an unsuitable geological history and which may be bounded in part by rocks in which oil is unlikely to be found or be underlain by relatively thin sediments, are in general unfavourable areas for marine exploration.

The second category includes the submerged periphery of the continental land-masses and larger islands, known as the Continental Shelf (defined by Pratt and Good, 1950, as the submerged periphery of the great platforms on which the continents stand in relief). Where the Shelf is known or believed to be formed of rocks which are a seaward extension of proved or potential oil provinces, then its prospects will be high. Examples are the shelves off the Californian and Gulf Coasts of the U.S.A. where commercially exploitable oil has been found. The Shelf off the North Carolina coast has been penetrated by boreholes in several places, but, like the boreholes drilled on the adjoining land, without success. The shelves formed or perhaps modified by the deltaic deposits of the world's great rivers form prospective territory as yet largely unexplored by drilling, with the exception of the Mississippi deposits in the Gulf of Mexico, where oil has been found.

Large areas of the Continental Shelf have, however, completely unknown or unpredictable prospects. This is mainly because the composition and origin are still matters for speculation, and no generally accepted explanation has yet been produced. This is not surprising, considering the inaccessibility of the subject. Extensive depth surveys have, however, revealed a considerable amount of topographic detail, and it is now known that the Shelf is seldom deeper than 112 fathoms, has an irregular surface where, however, differences in elevation are generally small, and has a very gentle overall gradient oceanwards of less than one degree (world average is 1 in 540); the width varies from less than one mile to about 700 miles. The oceanward edge of the Shelf is marked by a sudden increase in slope which persists, not without interruption, to oceanic depths, with a world average gradient of 1 in 15. This is now known as the Continental Slope and, like the Shelf, has an irregular surface frequently intersected by deep canyons, some of which cut into the Shelf also. The change in gradient between Shelf and Slope occurs at a fairly uniform depth throughout the world; the average is 72 fathoms (Shephard, 1948); but the 100-fathoms or 200-metres depth lines are more commonly adopted than the shallower figure to mark the edge of the Shelf.

At one time opinion favoured the idea that the

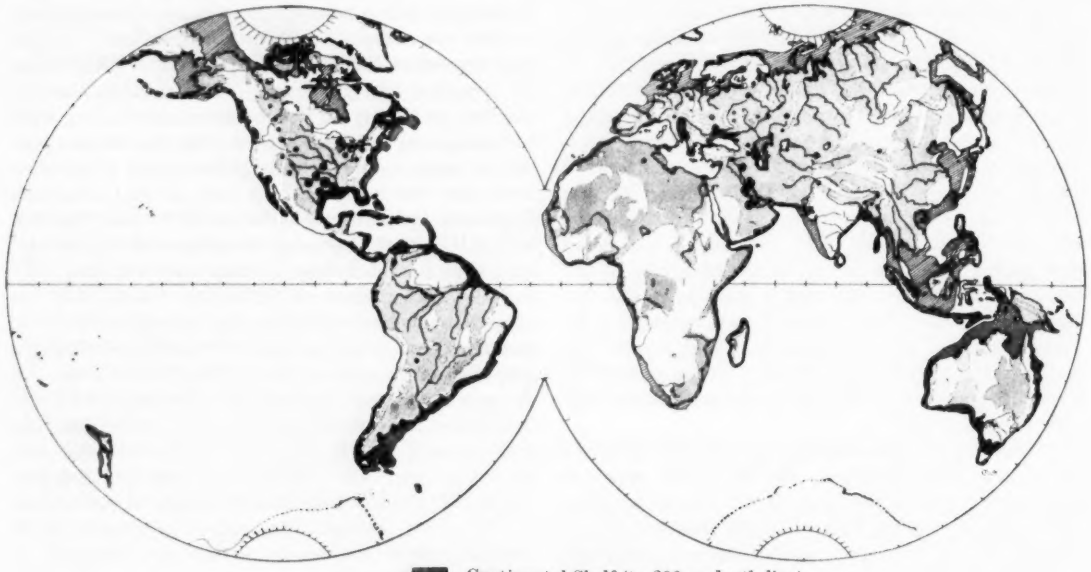

FIG 5

Continental Shelf was created by prolonged marine erosion of the continental edges, but it is now thought unlikely that this could apply to those areas where the Shelf is several hundred miles wide. A number of more recent theories, incorporating the results of geophysical surveys, attempt to establish a relationship between the formation of the Shelf, the nature of the earth's crust, and the permanence or impermanence of ocean basins. Most support is now given to those which invoke the deposition of sediment (clastic, organic, and chemical) on a substratum which subsided either as a result of increased overburden or by faulting between the continental landmass and the rocks forming the ocean basins, or by warping along the continental margin, associated perhaps with isostatic adjustment of the continent itself. Marine erosion, ocean currents, and glaciation must have contributed significantly to the formation, shape, and composition, but whether they should be regarded as of fundamental importance or as modifying agents is still uncertain.

Despite the differences of authoritative opinion which exist regarding the origin and nature of the Continental Shelf, there is difficulty in avoiding the conclusion that it is in part at least made up of a vast accumulation of sediments. Much, if not all, was deposited in an environment favourable to an abundance of marine life, but it does not necessarily follow that the Shelf was everywhere formed under conditions suitable for the generation and accumulation of oil. It is likely that the various modifying or formative agents, to which reference has already been made, will have caused a variation in oil potential as great as that of the sediments on land.

The area of the Shelf to the 100-fathom depth-line is approximately 10 million square miles according to Twenhofel (1932). If the Continental Slope is included, the area is increased to 25 million square miles. The total land area of the world is 54 million square miles, of which 45 million are covered by sedimentary rocks. Twenhofel has estimated that the volume of sediments in Shelf and Slope is probably between 70 and 80 million cubic miles and the volume on land about 45 million cubic miles. Pratt (1947) has calculated, after making an arbitrary allowance for rocks of unsuitable type and by a volume for volume comparison with the sedimentary rocks of the U.S.A., that the potential oil reserves of Shelf and Slope should be as much as 1 million million barrels, about three times the amount (production plus proved reserves) so far found on land. Such a figure would seem to be unbelievably high, but unless the volumes estimated by Twenhofel are wildly incorrect, then Pratt's method of calculation, even on the basis of comparison with the total volume of sediments on land, will inevitably yield high values. The figure for the Shelf alone would be between 150 and 200 thousand million barrels. This figure could, however, be substantially reduced, possibly by 25 to 30 per cent, if those areas of the Shelf which border unprospective land areas were omitted from the calculation.

The validity of the estimates and calculations may be questioned on such grounds as insufficient factual data regarding the composition of the Shelf and the use of comparisons between the uncertain and the largely unknown, but a scarcely unavoidable conclusion is that the potential oil reserves of the Continental Shelf and Slope or even the Shelf alone could be of a reasonably high order. Certain areas, such as the Gulf Coast of North America, can be expected with some confidence to justify this high rating. Other areas have much more speculative prospects, however, and in the light of present knowledge it would be an enormous gamble for any company to undertake the exploration of those areas in which, for example, the geological composition of the Shelf is likely to differ from that of the adjoining land.

Exploration results to date have gone a long way towards proving the potentially high reserves of such epicontinental seas as Lake Maracaibo and the Persian Gulf. The latter, by comparison with the Middle East oil province as a whole, could have reserves of the order of 50,000 million barrels. Consequently, exploration activity here is beginning to be intense.

Marine exploration is mainly a geophysical and drilling operation. The geologist's principal contribution is an initial general appraisal, based on the evidence provided by rock outcrops and by boreholes on the neighbouring land, and a later, more detailed appraisal based on marine geophysical surveys and the results from any wells subsequently drilled in the areas concerned. Attempts are being made to overcome the handicap imposed by the sea on the field geologist by employing trained aqualung divers to obtain "outcrop" evidence, such as rock-samples, dips, strikes, etc., from the sea bed. The British Petroleum Co. jointly with the Compagnie Française des Pétroles used this method at the beginning of 1954 (Cousteau 1954), at the commencement of exploration in the Abu Dhabi marine concession in the Persian Gulf. The sea bed, unfortunately, was covered by a mantle of recent deposits which effectively obscured most of the information being sought; recent work by U.S. companies off the Californian coast has been more useful. This will, however, be one of the drawbacks that can be expected to occur frequently, but even under ideal conditions of no obscuring cover, light currents, and calm weather over protracted periods, the method cannot be expected to yield other than a very general picture of the geology. Underwater photography and shallow coring from ships are other methods used to obtain information, but they too have a restricted value.

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III. GEOPHYSICS

By A. B. Malone

We have heard that a large proportion of our future oil reserves are likely to be found in marine areas. For obvious reasons, in most parts of the world our geological knowledge of the sea bed is very meagre, and to obtain the necessary information to enable us to locate potential oilfields we can hardly depend on such direct evidence as may be collected by divers, or dredged from the sea bed, but it is necessary to employ indirect geophysical means. The principles and main methods of geophysical exploration on land are well known and have been established for many years, and fortunately it has been possible to adapt them to marine areas. In some cases the adaptation has not been easy, but the change has been facilitated by a transition phase as the equipment was first modified for use in swamps and shallow inland waters.

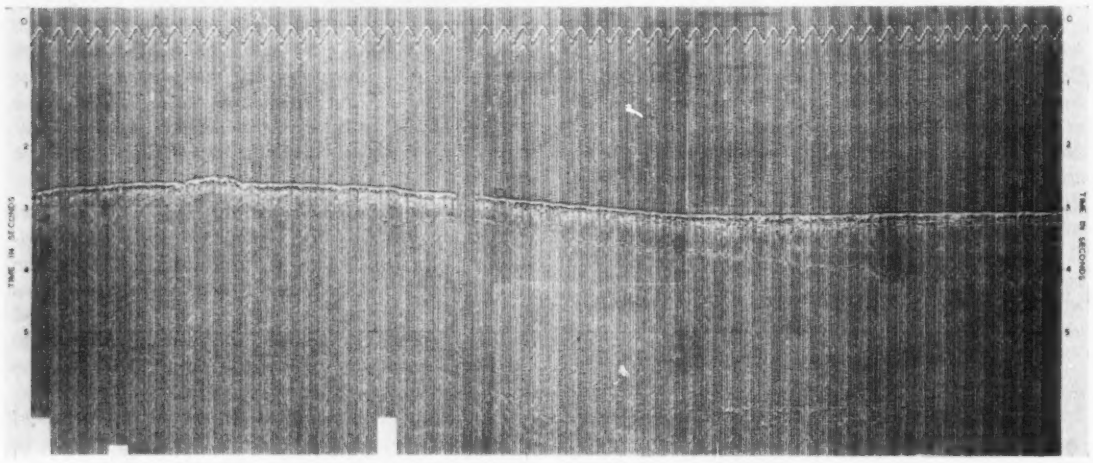


FIG 6
A VARIABLE AREA CROSS-SECTION SHOWING STRUCTURE BELOW THE SEA BED
(by permission of Shell Petroleum Co. Ltd)

As on land, the three main oil prospecting tools are magnetic, gravimetric, and seismic. The land magnetometers used before the last war have largely been superseded by the airborne magnetometer, which, though initially developed for geophysical purposes, found its first practical application in the detection of enemy submarines. Since then it has been widely used as a reconnaissance aid over large areas of swamp, desert, or forest where the stratigraphy was obscured. The method has the great advantage that by air large areas difficult of access can be covered in a very short time, and obviously such a technique can immediately be applied to marine surveying. Since one of the principal causes of variation in the magnetic field is changes in the magnetization of the igneous rock complex which underlies the sedimentary rocks, it is possible to use these magnetic measurements to determine the approximate thickness of the sediments and thus indicate basins which may be of interest to the oil prospector. More, it may result in a great saving of money, time, and effort by indicating where sedimentary thicknesses are small and thus where it would be useless to introduce costly seismic methods. Unfortunately, such clear-cut results are

not always obtained. In some cases the basement rocks are rather uniformly magnetized, and are of such low susceptibility that they do not give rise to the changes in magnetic field intensity necessary for depth calculation.

Where the area is sufficiently large, the method is much cheaper per mile of traverse than a land survey, but where the area is too small to warrant the expense of an airborne survey, results can be obtained by towing the recording element behind a ship. A recent development of an instrument suitable for this purpose is the Varian water bottle magnetometer, in which the determination of the frequency of free precession of the protons in a sample of water around

FIG 7

DETECTORS ARE SPREAD OVER DISTANCES OF ½ MILE AND TOWED BEHIND THE SHIP IN A FLOATING CABLE

the earth's field enables that field to be calculated. This instrument has led to a reduction in the complexity of the equipment used in magnetic prospecting and to the development of portable units which can be carried by one man.

The gravity meter has long been in favour as a reconnaissance instrument on land. When first applied to marine work the gravity meter was set up on platforms on a long tripod standing on the sea floor. Some of these tripods were large structures standing 30 ft high, built of tubular steel, and transported by towing as a hydroplane behind the survey vessel. Various varieties of diving chambers were also used in which instrument and observer were lowered to the sea floor. In due course, gravity meters were developed which could be lowered to the sea bed and worked entirely by remote control. With this type of instrument many successful surveys have been carried out, and it is reported from the Gulf of Mexico that more salt domes have been found at sea than have been discovered on land by the normal gravity meter.

Up to the present a marine gravity survey is subject to delays due to bad weather and is rather slow and expensive as compared with a similar survey on land. A meter which could be used on board a vessel would enable gravity surveys to be carried out much more rapidly and thus take advantage of the better weather conditions. Meters have now been used successfully in submarines and are being tried on surface ships. It has been reported very recently that these trials have been successful, and we may expect that a large increase in marine gravity surveying will follow. It would clearly be of even greater advantage if an instrument could be developed for use in an aeroplane, but here we come up against the very difficult problem of overcoming the rapid changes in acceleration which are encountered in flight.

The most useful and accurate geophysical tool has been the seismograph. Two methods are employed, reflection and refraction. The principle of the former is similar to that of the echo sounder employed at sea for determining the depth of water. Shock waves from an explosion are reflected from the underground rock layers and are registered at the ground surface by several groups of detectors spread over a distance of about half a mile. From the records obtained it is possible to determine accurately the time of travel of the reflected disturbance and thus to calculate the depth and dip of the reflecting surfaces. When applied to work in shallow water attempts were made to plant the detectors in the mud of the swamps, or sink them to the bottom of the lakes. These methods were successful but time-consuming. When more open waterways were reached, it became possible to mount the phones on a floating cable and tow the spread from one observation point to the next, with a considerable saving of time. Some methods use the ordinary land velocity type detector, which requires to be orientated nearly vertically and thus has to be mounted on paravanes. Other methods use pressure-sensitive detectors, which are independent of the orientation and thus can be mounted in the tow line or loosely suspended from it. It is found that by slacking off the tow line for a few seconds, the spread of geophones becomes sufficiently stationary in the water to enable the shot to be fired and recorded successfully. Thus the parent ship does not need to be stopped. The time between shots ¼ mile apart may be as low as two minutes, and by this means, even allowing for difficulties through bad weather, outputs ten to twenty times those on land may be achieved. Marine reflection work is now so fast and relatively inexpensive that sometimes, even when only land drilling is planned, it is better to get information by carrying out an offshore or estuarine survey first before tackling the land itself.

The marine seismic method has the advantage that there is little limitation on the size and weight of equipment that can be carried, and therefore up-to-date forms of recording can be used. It is now customary to employ magnetic recording and specialized equipment for playback purposes and thus

produce long record profiles on which the data can be presented in the newly-developed forms such as variable intensity, variable area, and so on. This overcomes to a large degree the problem of coping adequately in a reasonable time with the great output of a marine survey. By adapting the recording of the seismic data on tape to a form suitable for operating modern digital computers, it may yet be found that the large teams of geophysicists employed in the past in analysis and interpretation are no longer needed, and the results of marine shooting may be obtained within a few days instead of months.

Such marine surveys will furnish us with valuable information from depths between, say, 500 and 20,000 ft, the seismic frequencies involved varying from about 200 to 20 c.p.s. By employing higher frequencies we can obtain better resolution of rock beds at shallower depth, and an interesting development in this direction has been that of the so-called Sonoprobe. This is an instrument which employs an acoustic source having a frequency around 4000 c.p.s. and with which information can be obtained from the sea bed itself and perhaps down to 200 ft below. It can furnish a profile which indicates, for example, the thickness and form of the soft sediments lying on the sea floor and of the outcropping harder rocks below. Quite clearly, such an apparatus can be of considerable value when planning the location of piles for structures such as jetties, drilling platforms, and so on.

Refraction seismic work has very considerable application on land, though its use in marine areas has hitherto been restricted. In this technique observations are made on the shock waves from an explosion which are refracted by the rock layers and travel along these layers. Rocks of different types have characteristic velocities, and these velocities may be determined from measurements of the times of arrival of the shock waves at detectors positioned along a traverse. When the rock layers are dipping the apparent velocities as measured differ from the true velocities of the rock media, and this affords a means of dip determination. The method has been employed at sea by oceanographers to secure information about the earth's crust, thus supplementing their information obtained through the agency of natural earthquakes. Perhaps one reason why this method has not been employed very much for purposes of marine oil exploration is the fact that the reflection method has had such spectacular success.

Marine and airborne geophysical surveys naturally require fast, accurate methods of survey positioning. Such methods are available as a result of remarkable developments in electronic types of surveying such as Gee, Shoran, Loran, Decca, Raydist, and Lorac. The British Decca system is a particularly valuable one for this purpose, and Decca chains are being installed in a number of areas of oil interest for geophysical and other purposes relevant to the oil industry. In the Decca system three transmitters are set up in locations which are accurately positioned at strategic points in the area. Transmitters A and B transmit continuous radio waves on the same frequency and transmitter A also transmits a radio wave on another frequency which is in phase with that transmitted on a similar frequency by transmitter C. When the difference in the paths from transmitter A and B to a receiver on board ship is an integral number of wavelengths, the two signals will arrive in phase and reinforce each other: elsewhere the signals will interfere. The locus of points, the difference of whose distance from the two fixed transmitters is constant, is a hyperbola, and thus the transmission from these two transmitters establishes a radio net over the area which is divided into lanes by a series of hyperbolæ of signal reinforcement, the foci of the hyperbolæ being at the transmitters A and B. Likewise, transmitters A and C radiating on another frequency set up a second set of hyperbolæ which form a grid with the first. The position of the ship in the network is given by its Decca co-ordinates within this grid, and may be continuously recorded as the ship moves along a traverse.

Using this network, it is also possible to plot the position of the ship continuously on a graph, or to run the ship along a pre-determined course. Once the phase counters are set up on the correct lanes, they will continue to record any change in co-ordinate position correctly, but if switched off for any reason, it is important that they should be restarted correctly. Hence it is usual to set up a marker buoy or to return to a known position at night. The necessity for this can be eliminated by setting up a fourth transmitter for " lane identification."

The marine method of seismic reflection is obviously one calling for great expense in ships and equipment, and it needs a high degree of skill, co-ordination, and team-work. It also needs thorough advance preparation, installation of costly surveying equipment, and expenditure of large quantities of explosive. It can easily be held up by bad weather, and locally depth and bottom conditions may cause such a channelling of energy that all reflections are lost—a phenomenon known as " wave-guide." Nevertheless, in general it has very many advantages over land work. Despite the initial expense, records are produced so fast that the cost per mile of survey is only about one-third of that on land. Traverses can be laid out to provide a uniform overall coverage pattern, and thus less shooting is required than on land, where traverses cannot usually be laid out to such good advantage. Corrections are generally smaller and results better and more accurate. An area requiring years of work on land can be surveyed in a few months, and thus the costs of the whole venture can be reduced. The navigation networks can also be used for other parts

of the company's activities, *e.g.* general mapping of the area, and thus reduce costs.

By employing such geophysical techniques, with ancillary aids, a contour map can finally be prepared which demonstrates the undulations of various stratigraphic horizons below the ocean bed. Features of geological interest can therefore be defined for purposes of a drilling location. It must be remembered, however, that no geophysical method has yet been defined which will furnish a reliable direct indication of the presence of oil: at best it can only define those structural features which are favourable to oil accumulation, if oil in the area exists. Confirmation of the presence of an oil reservoir can only be given by the drill.

IV. DRILLING BARGES

By W. A. Roy

There are a very large number of types and varieties of structures or barges now being used for submarine exploration, and new types are constantly appearing. It has already been mentioned in this symposium that the majority of underwater projects have been undertaken in the Gulf of Mexico or in Lake Maracaibo, and for this reason practically all of the design work has originated in the U.S.A.

The large number of different designs which have been produced is only to be expected because the operation is comparatively new and its growth has been so rapid that many different groups of people have been working concurrently and their ideas of what is the correct solution are very different. Again it should be remembered that the multiplicity of types have been designed in order to comply with a widely different range of conditions, from shallow and protected waters to deep water in the open sea.

If inshore exploration is contemplated the selection of the type of equipment to be adopted is much more simple than if the work is going to be carried out in deep water in unsheltered conditions. While this is true, however, a very complete analysis of the requirements should be made even for inshore work, because the capital expenditure is of necessity high and a detailed examination of every aspect of the problem, not just the major items, will be amply repaid.

The governing conditions for the selection of the most suitable type can be briefly stated as follows:

1. Depth of water likely to be encountered.
2. The estimated drilling depth.
3. Whether the drilling is to be carried out in sheltered waters or in the open sea, in which case the structure must be designed to withstand severe storm conditions.
4. Whether the exploration is being carried out in an area in which supplies of materials, spare parts, etc., can be quickly obtained or whether the area is remote and these are difficult to obtain.
5. The type of bottom which is likely to be encountered.

There is no generally accepted classification for the various types of offshore equipment. For the purpose of this paper the following groupings have been made, but it is desired to make it very clear that there is a wide range of designs in each group.

(*a*) Self-contained fixed platforms.
(*b*) Fixed platforms serviced by tender.
(*c*) Self-contained floating barges.
(*d*) Self-contained mobile units with submersible pontoons.
(*e*) Self-contained, self-elevating, mobile type.
(*f*) Drilling in very deep water from surface vessel.

Before the selection of any particular type of equipment can be made it is generally essential that meteorological, oceanographical, and sea bed surveys are made. In certain areas where offshore operations have been practised for a number of years excellent facilities for making these surveys now exist, but in other locations which are just being opened up this is not so and the compilation of information which is of any real value will take a considerable amount of both time and money.

There is a growing awareness among operating companies of the need for a central meteorological and oceanographical information system to which all can contribute for the common good, as it is realized that it is only by that means that reliable information can be given to the design engineers which will enable lighter and more economic structures to be produced.

Fixed Structures

In some of the first submarine exploration work which was carried out nearly twenty-five years ago, fixed structures were used, and the platform on which the steam-driven drilling equipment was installed was carried on creosoted wooden piles. One of the first of this type had a platform of dimensions 180 ft × 220 ft and it stood 15 ft above water level. The same layout of equipment as for a land-based rig was adopted, and this is very uneconomical for marine operations, where every square foot of deck space has to be utilized to the full, consistent with all necessary safety precautions being met. The next development of the fixed structure, still using wooden piles, was to arrange the drilling equipment on different levels and by so doing reduce the surface area of the platform and its cost.

The wooden piles were gradually replaced by steel piles or reinforced concrete piles, and this enabled structures to be constructed which could operate in deeper waters.

For exploration work the fixed structure has the very obvious disadvantage of lack of mobility. The initial expenditure is high and the cost of moving to another site is considerable. Any operation of this kind is one in which the weather can play a very big part, and despite the fact that experienced teams of erectors with heavy mobile lifting equipment have

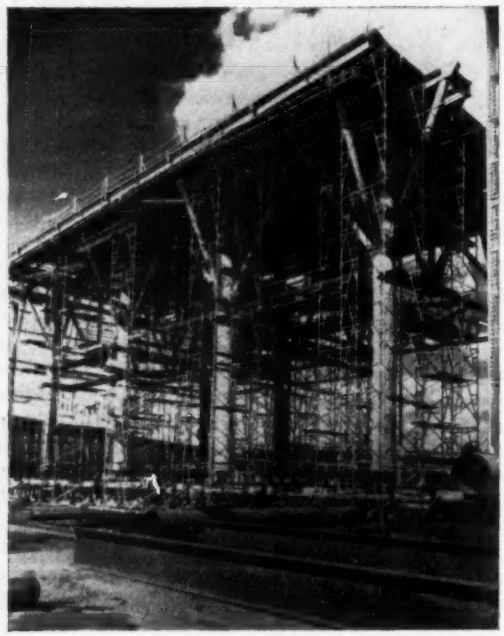


Fig 8

been got together and are now an integral part of the offshore operation, many very expensive days are lost each year due to this cause.

Many large fixed-type tubular structures have, however, been made for the exploitation of known fields, being used not only as drilling rig platforms from which a number of holes can be drilled, but also in groups on which separators, pumps, and other production equipment are supported. These large units are designed to withstand severe storm conditions, are generally self-supporting for considerable periods, and carry accommodation for up to fifty people. They fall outside the scope of this paper, but in order to give some idea of their size and the type of construction facilities required, one with platform dimensions of 90 ft × 140 ft is illustrated in Fig 8. This particular platform weighs 1032 tons and is supported on sixteen 30-inch diameter steel piles 195 ft long.

Tender Supplied Type of Structure

The high cost of the large fixed structure for exploration work brought about the introduction of the smaller type of fixed structure, which can be serviced by an attendant marine craft of some kind, and which could be much more easily dismantled should a dry hole be found.

One of the first of this type of structure had a total deck area of 38 ft × 76 ft and stood 20 ft above high water level. The main part of the deck, which carried the derrick, drawworks, and rotary table, was supported on sixteen 24-inch o.d. steel piles which were driven 100 ft into the sea bed.

The tender was a converted naval craft of some 2300 tons displacement—260 ft long × 48 ft beam—and this carried the accommodation for the crew and also fuel, fresh water, the cementing unit, mud tanks, and drilling consumables.

To prevent damage to the structure by the tender during bad weather, three creosoted timber dolphins were placed between them, and these dolphins were also a considerable help in mooring the tender. The mooring of any marine craft in unsheltered waters is never an easy operation, but in the case of a drilling tender it is particularly difficult because it must be kept relatively stationary to the fixed structure which carries the derrick. Some locations are easier than others, depending on the nature of the sea bed. Where it is soft, conventional types of anchors can be incorporated into the system, but where hard smooth bottoms are encountered they drag and have very little value.

Most tenders now used have to be towed out to location and towed off during bad weather, but some are self-propelled and are able to ride out a storm in the vicinity of the structure and so be quickly connected up to it when calm weather returns, thus saving much valuable time.

Many of the original tenders were converted naval craft, and while their use was economical as far as initial expenditure was concerned, the layout of the equipment was not always satisfactory. Now, tenders are specially designed for the job, and in some cases the diesel engines are used to propel them and also supply the power required for the drilling operation.

The combination of the small fixed structure and the tender, an example of which is illustrated in Fig 9, has been widely used, a recent estimate being that between 80 and 90 per cent of the marine wells drilled have been done in this way. The main objection to it is that it is not independent of weather conditions, as all tenders have to break away in bad weather and during this time all the drilling operations cease, but the high hourly cost of running the outfit still continues.

Self-Contained Floating Barge

In certain locations, particularly around the coast of Louisiana, the water table is only a few feet below

ground level and there are larger areas of swampy territory which make the laying of roads suitable for the transportation of heavy equipment a very difficult and costly business.

To get over this problem the idea of using self-contained shallow-draft barges was conceived, by means of which approach to this sort of territory could be made from the seaward instead of the landward side.

Many of the early examples of this type of equipment merely consisted of two pontoons braced together with a space between them which provided the drilling slot, the drilling equipment being carried on a

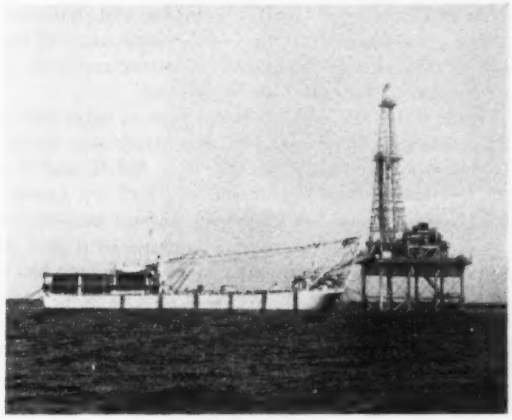


FIG 9

substructure built across the two pontoons. Most of the other equipment, such as the mud pumps, the mud tanks, and the diesel electric generator equipment, was carried on the deck of the pontoons, and provision for storage of fuel and water was made below deck.

Arising out of the operating experience which has been gained, an almost standard pattern for this type of barge has emerged. It is either of two- or three-deck construction, and the drilling derrick and pipe rack are placed on the top or on a substructure. Other essential equipment, such as the generating equipment, pumps, etc., are placed below the main deck, and supplies of fuel and fresh water are carried in tanks built into the hull of the barge. Air-conditioned accommodation for between twenty and twenty-five people is provided, and it is always located at the opposite end of the barge from the derrick.

A recent example of this category of barge has been designed to operate in water depths from 6 to 21 ft. It has overall dimensions of 140 ft × 60 ft and is of the two-deck, single-pontoon type The barge is placed on location by flooding the pontoon till it settles on the bottom and pumping it out when it is desired to move to another site. The pumps for this purpose are carried on the lower deck of the main structure. It has a diesel electric drive to the draw-works, two 5-ton service cranes, and air-conditioned accommodation for twenty people.

While scouring is not the serious problem on inshore barges that it is on the large submersible mobile units, provision is made in this particular case to have projections on the bottom of the pontoon which key themselves into the soft bottoms where it is used and act as an additional safeguard against movement.

Large Mobile Units

As the leases for inshore areas became more and more difficult to obtain, large mobile units were developed which could take advantage of those offered for deeper waters.

This class of unit can be divided into two main types:

(1) The submersible type.
(2) The self-elevating barge type.

This phase of the operation presented the design engineer with new problems to solve. When the operation was limited to sheltered waters, the principal forces which had to be taken into account were:

(*a*) Those due to the wind.
(*b*) Those due to the static load of the platform and its equipment.
(*c*) Those due to the derrick loading during drilling.

Now wave forces had to be included, and the accurate assessment of their values is a very complex problem. A number of theories exist by means of which they can be computed, but unfortunately they give divergent results because the basic assumptions regarding wave form and allowances for the drag effect are not the same in each case. Because of this, considerable care must be used in the selection of the one to be used for a particular set of conditions, and this decision is best left to oceanographic consultants after a survey of the sea bed and wind and wave conditions has been made by them.

A great deal of work has been carried out on this subject during the past few years which has resulted in more reliable data being made available, but despite this fact, expert advice should always be obtained for any new location, because it is essential to both the type selection and the design of the unit which will be used.

Some indication of the extent of the wind and wave forces may be of interest, and the figures below apply to the elevating barge shown in Fig 12.

The barge dimensions are 196 ft × 106 ft × 15 ft, and the all-up weight in the drilling position is approximately 4750 tons. The specification demands that the structure should be able to successfully withstand wind speeds of 100 m.p.h. and be able to operate in a water depth of 80 ft, where the wave conditions,

representing the 100-year storm possibility, were as follows:

Period	9·4 sec
Length	430 ft
Maximum height	35 ft
Crest elevation	107·5 ft
Trough elevation	72·5 ft
Speed	45·7 ft/sec

For these conditions the total wind pressure amounts to approximately 130 tons. The maximum wave force on any one spud leg is 64 tons, and under the worst conditions the total wave force to which the structure can be subjected is 84 tons, resulting from a force on two legs of 127 tons in the direction of travel of the wave and a force of 43 tons on the other two legs in the opposite direction.

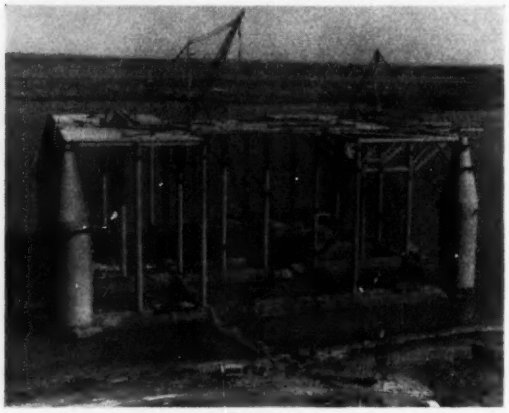


FIG 10

It is not easy to draw a sharp dividing line to indicate the limiting conditions of either the submersible or self-elevating type of mobile unit because both have been designed to operate in water depths of up to 100 ft, 125-m.p.h. winds, and sea conditions where a 40-ft wave is a fifty-year probability. One difference does stand out, and that is while the self-elevating barge can operate on any type of sea bed, the submersible type is limited to soft bottoms. Further to this an analysis of the design specifications of more than seventy mobile units indicates that in general the self-elevating type is operating in deeper water than the submersible type.

Submersible Type

This type can be sub-divided into two classes:

(*a*) The pontoon type.
(*b*) The fixed-height platform type.

In type (*a*) the main platform is towed out to site on pontoons through which locating piles can be driven. The piles are in some cases only entered into the sea bed by hydraulic jacks, and the weight of the platform is applied to diagonally opposed piles to obtain the necessary penetration. In other designs the piles are completely driven by hydraulic jacks mounted on the main platform. After the piles have been driven, the pontoons are flooded and sink to the sea bed, thus forming a structure in which the piles are tied above water by the platform and at the sea bed by the pontoons.

The problem of scouring is one which is always present with large pontoons used in this manner. Where two pontoons are used to support the structure, the scouring effect is not the same on each, because under certain tide conditions one of the pontoons can act as a shield to the other. This can result in unequal loading conditions being produced, sometimes with disastrous results. The effect of scouring is receiving the close attention of both structure designers and oceanographers, and this co-operation will doubtless lead to a fuller understanding of the basic cause of the problem and the development of better methods of overcoming it than are used at present.

Fig 10 shows one of the second type of submersible unit under construction. In this particular design the platform dimensions are 240 ft × 202 ft, and it is supported 100 ft above the lower hull by twenty tubular columns of 3 ft 6 inches diameter and two of 4 ft diameter. The lower hull consists of a grid of tabular members 14 ft 6 inches diameter which has a built-in skirt to resist side motion and scouring. At each corner of the grid are conical-ended cylinders, the lower 65 ft of which are 22 ft in diameter. These cylinders are used as stabilizers during the lowering and elevating operation. The approximate total weight of the unit on the bottom with the grid system flooded is approximately 4500 tons, and it can operate in 70 ft depth of water.

Another version of this particular design has recently been constructed which embodies eight locating spud piles, the supporting columns acting as jackets for driving the piles. The actual driving and retracting is carried out by means of hydraulic jacks carried on the main platform.

Fig 11 shows another example of the fixed-height submersible type being towed to location. This illustration serves to convey some idea of the problem which is presented in the towing of these large structures. The number of companies who have both the equipment and the knowledge required for this operation is very limited, especially where bad weather conditions are likely to be encountered. It also illustrates the somewhat unusual gantry-type service crane which is used for service and supplies, and in addition the tapering of the bottom hull to avoid scouring can be seen.

Self-Elevating Type

This type falls easily into two groups:

(*a*) Those using air or hydraulic power for elevating.
(*b*) Those using electric power for elevating.

In each case the platform or barge is designed to be capable of being towed to site floating in the water, and as such must be able to withstand storm conditions in this position. When the hull is in the elevated position it must be able to support its own weight in addition to that of all the equipment and stores and the drilling load. To achieve this a very rigid design is adopted, which up to the present time has taken one of the following three forms:

(1) A rectangular flat plate hull stiffened in the egg-box pattern by lateral and longitudinal bulkheads.

(2) A triangular hull of corrugated plate construction.

(3) A rectangular hull of large diameter tubes braced by the upper and lower decking. In this design the tubes are used for the storage of drilling water, potable water, and fuel oil.

In all types the derrick is carried on a movable substructure which, in the drilling position, is placed over a slot built into the hull of the barge. In this position the derrick floor is usually between 40 and 60 ft above sea level, and before lowering the barge it is necessary to retract the whole of the derrick structure inboard in order to clear the wellhead and conductor tube and allow the barge to be moved away from the well.

The elevation in each case is carried out on caissons or spud legs, the number of which varies and is dependent on the sea bed conditions. These spud legs are usually either of solid tubular or latticed design, and in some cases reach lengths of 220 ft; the lattice design, which reduces the effect of the wave forces, is used in both the rectangular and triangular form. Where soft bottoms are encountered, the caissons generally have large tanks or "feet" up to 30 ft diameter fitted to them in order to reduce penetration and give a more even load distribution.

FIG 11

The power for elevating and lowering is derived from either hydraulic or air jacks or from a series of electric motors driving pinions which engage in racks built into the spud legs. The elevating and lowering speed is very important in the design and operation of this type of platform, and speeds of up to 70 ft/hour with all-up weights of in excess of 4000 tons are now being achieved. The necessity of the high elevating and lowering speed is that the most dangerous period of the operation is when the barge is just clear of the water and is still within the range of a wave which might be encountered during the lifting or lowering operation. As the exposed bottom and side area in many cases exceeds 20,000 sq. ft., it will be obvious that very dangerous conditions can be produced during this period. The lifting speed is proportional to the elevating power applied, and whether the need for speeds as high as 70 ft/hour can be justified is a doubtful one. There is no doubt, however, that it is very necessary to get clear of the danger zone quickly, and in this connexion the existence of reliable meteoro-

logical data for the lifting and lowering period can greatly minimize the risk incurred.

Drilling from Surface Vessels

Surface vessels are now being used for carrying out drilling in very deep waters up to 650 ft. This development is still, however, in an experimental stage, and while it is not proposed to make any further reference to it now, one particular type of drilling equipment will be mentioned later.

Layout and Equipment

There is no accepted pattern for the layout of the equipment on either type of mobile unit, the one common feature being that, as in the case of large fixed units, the accommodation is placed as far away from the derrick as possible. Great ingenuity has been displayed in the disposition of the large amount of equipment that has to be carried, with the ever-present aim of effecting the maximum possible use of the space available and by doing so achieving reductions in the overall sizes and costs.

Diesel electric drives to the drawworks are becoming more common. Although the initial expenditure incurred in their installation is greater than with the standard diesel engine drive via transmission units, this type of drive permits of much more latitude in the layout of equipment, and consequently better use of the available space.

It is now an accepted trend to put as much of the equipment under cover as possible, but care must be taken to see that in doing so easy access is available to all equipment which requires periodic maintenance and that all electric cables and hydraulic piping are distinctively marked. A few hours' thought given to these problems during the design stage can save many days of hard and unnecessary work later on.

In the unit shown in Fig 12, air-conditioned and sound-proofed accommodation is provided for fifty people arranged on two different deck levels. It is placed below the helicopter landing deck, which serves to provide shade from the sun in very hot locations.

The total installed horse power is 2800, of which 1200 is used for driving the electric generators, which are housed in a separate compartment, along with the necessary switchboard and control gear. The total connected load is about 1200 h.p., but the average demand is in the region of 400–500 kW.

The drill rig and drawworks in this case are driven by four diesel engines, the transmission unit being arranged in two tiers to save deck space.

350 tons of drilling chemicals are stored in three hoppers carried on the main deck, and a conveyor system connects them to the mud tanks, which are also situated there. Fuel, drilling water, and potable water are carried in tanks which form part of the hull construction.

A flash evaporator is installed below deck in the pump room, and this is capable of supplying 600 g.p.h. of fresh water. It uses the waste heat from the diesel engines, and when these are not running or when the load demands are low, an additional source of heat is available in the form of an electric boiler. On some other units vapour compression distillation units are used, but where comparatively large amounts of waste heat are available there would appear to be a good case for using the waste heat evaporator type of equipment for the production of fresh water supplies.

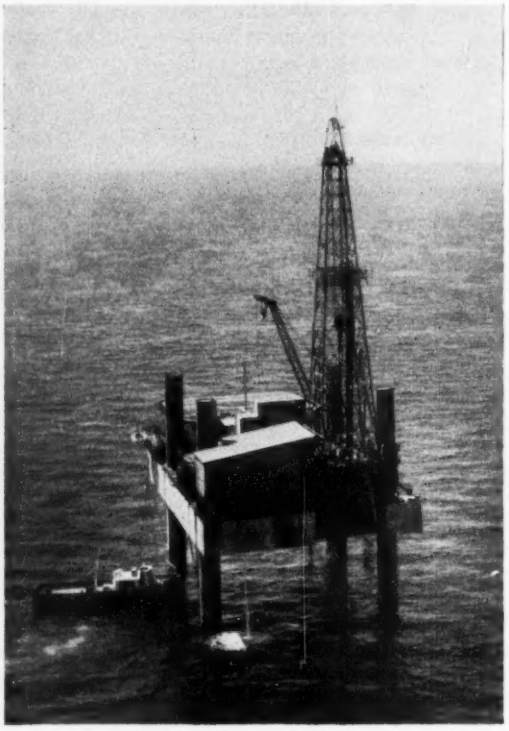


Fig 12

An adequate communication system between the offshore unit and its base is essential to the success of any project. On the unit being described, there is radio and telephone equipment by means of which it can keep in contact with its service marine craft and helicopters in addition to its shore base. An internal telephone installation is also provided, the instruments situated near the derrick being of the flameproof type enclosed in soundproof housings.

On all rigs considerable attention is now being given to making the standard of the accommodation for the operating crews as good as possible. The psychological aspect of the offshore operation is becoming more

appreciated, and it is now realized that good accommodation, food, service, etc., play an important part in the cost of drilling a hole in the long run.

As these mobile units operate at between 30 and 40 ft above the water, service cranes have to be used to bring all supplies aboard from marine craft, and for this purpose three types of crane are used:

(1) The gantry type shown in Fig 11.
(2) The fixed-position rotating type.
(3) The tracked crawler type.

Each type has its supporter's individual preference, rather than any fundamental advantages being the general basis for selection.

Mention will be made in another section of this paper of the high cost of the construction and operation of these large mobile units, and there is no doubt that considerable pressure will continue to be exercised on the designers to reduce the overall sizes still further, and in this connexion efficient layout and maximum utilization of space can make an important contribution.

Servicing of Offshore Operations

One of the most important factors in the offshore operation is that of supply. The success of any such project depends largely on continuity of working, and this can be achieved only if provision is made to ensure that adequate quantities of all kinds of necessary supplies are available when required.

In the Gulf of Mexico and in Lake Maracaibo the offshore operations have brought into being a large number of new industries which deal solely with this problem. When it is realized that hundreds of self-contained units, ranging from the small fixed platform to the large mobile barges, have to be kept supplied with food, fuel, chemicals, equipment spares, drilling consumables, etc., some idea of the magnitude of the operation can be obtained.

Large numbers of marine craft, such as barges, tenders, fast launches for crew changing, and many other types of special craft, are in continuous operation 24 hours per day from shore-based establishments which are grouped around the operating areas. Also grouped around these areas are fully-equipped service depots which have been set up by the principal equipment suppliers, and from these prompt deliveries of spare parts can be obtained in cases of breakdown.

This comprehensive supply system has had its effect on the design of structures operating in sheltered waters in so far that they can be made smaller because of the reduced demands for storage space.

When the operation takes place in unsheltered waters many of the supply services cannot be taken advantage of because of the risk of isolation during storm conditions. This has meant that the big outfits have to be completely self-sufficient for periods up to three weeks.

The supply industry has grown very rapidly during the past few years, and specialist services can now be obtained, some examples of which are given below:

(1) The supply of ready-mixed mud, which is transported in a specially designed barge.
(2) The supply of hot meals on a 24-hour per day basis.
(3) The shotblasting and repainting of structures *in situ*.

Helicopters are used extensively for routine executive visits and the supply of urgently needed spares, and when bad weather conditions prevail they are used for crew changing and the transportation of supplies which would normally be undertaken by marine craft.

In cases where the operation takes place in a remote area where the above-mentioned facilities are not available, a base will have to be established from which it can be maintained. When this is, of necessity, on shore, harbour facilities for the marine service craft, an airstrip, workshops, stores areas, etc., will have to be provided in addition to adequate housing, office, medical accommodation, and recreation facilities for the base personnel and operating crews.

Another important aspect of the supply service is the effect it has on the morale of the operating crews. Where the service is bad it is extremely difficult to avoid a feeling of frustration and isolation prevailing, but where the service is good and adequate supplies of materials, good food, newspapers, magazines, etc., are available a great deal of the irritability, etc., which occurs when groups of men are living together in restricted spaces can be avoided.

Corrosion

One of the greatest enemies of the offshore operation is corrosion. The annual loss due to damage from this cause on all the outfits now operating is a very considerable figure, and in many of the earlier designs little or no attempt was made to combat the problem. This can probably be attributed to lack of appreciation, first, of the seriousness of the problem, and secondly, of the comparatively small expenditure which is required to effect large savings in the long run.

When it is considered that on certain locations the annual corrosion rate in the splash zone areas of unprotected structures can reach values of 25 thousands of an inch per annum, some indication of the total wastage can be envisaged.

The generally accepted method of preventing corrosion is by the application of paint, but this is often applied without sufficient care or to uncleaned metal surfaces, and if this is done it offers little or no permanent protection. Before a paint job is done the surfaces of the metal should be either gritblasted or shotblasted or given some form of phosphate treatment. If either of the above methods is not available, some form of treatment to remove the mill scale,

followed by mechanical cleaning, should be the minimum requirement.

During the past few years rapid strides have been made by the paint manufacturers in the production of good protective coatings. The first group of these was the vinyls, which have excellent resistance to sea water, and a 5/6-coat system is generally recommended. The newest group of coatings are the epoxy resins, and here again good protection can be obtained if the application is carried out on a properly cleaned surface. It is also possible to obtain 100 per cent inorganic zinc coatings which contain no oils or resins and which, if applied to shotblasted surfaces, will give, it is claimed, complete protection for a number of years. One large company which has made an accurate appraisal of the damage due to corrosion on its equipment is not only going to take care of the structures, but is going to galvanize all machinery installed in exposed positions.

The submerged areas would appear, at first sight, to be the most difficult to deal with, but fortunately this is not the case, because permanent protection can be given by the use of a cathodic protection system. When it is considered that some structures have up to 50,000 sq. ft. of submerged surface the importance of adequate protection will be appreciated, especially in fixed structures where no routine maintenance can be carried out as in the case of mobile units.

There are two types of cathodic protection system:

(1) By the use of sacrificial anodes of magnesium or zinc.
(2) By the use of impressed d.c. current.

Where no electric power is available, sacrificial anodes are used and are generally designed to give two years life before requiring replacement. Where electric power is available the impressed current method is becoming more popular because the carbon or lead anodes used last for a very long time and also the density of the current, usually about 4 milliamps/sq. ft., can be controlled from the platform deck.

The most difficult area to protect is the splash zone, and this is the worst affected area. It is general practice now, where designs permit, to use some form of plastic wrap-over, an epoxy resin or zinc sprayed coating, but the use of Monel metal as a sheath for all parts of the structure in this area is being strongly advocated. The initial cost is high, being in the neighbourhood of 20/-*d.*/sq. ft., but it is claimed that even this high cost can be justified by the results obtained.

Where the depths of water are not great and the locations are sheltered, reinforced concrete has been used with considerable success, particularly in Lake Maracaibo. Experiments with aluminium are also being tried there, and recently two large pile jacket structures have been built and are now on location.

Safety

All the hazards which apply to drilling on land are present in marine drilling, but they are generally accepted as being more serious because of the confined space which, of necessity, prevails. In addition to these hazards there also exist other dangers which are connected with a marine operation of this type. Two examples are indicated below.

(1) The transference of personnel from service craft to the drilling structure.
(2) Confined space and difficult deck conditions.

Many accidents have occurred during the transfer of operating crews during bad weather, and a number of methods have been tried to eliminate this hazard. The most successful of these would appear to be the use of a collapsible rope cage to which the personnel can hold on while it is being lowered by the service crane, any baggage they have being placed inside the cage. When the rope cage lands on the deck of the service craft, it collapses and so prevents the personnel being hurt, as was the case when a non-collapsing type of cage was used.

With regards to the second item, it will be appreciated that it is far from easy to keep the decks clear of mud, etc., during the drilling operation and a high proportion of minor accidents occur because of slipping. Anti-slip treads should be used wherever possible, all stairways should be erected with as flat a pitch as possible, sharp corners should be removed everywhere, and as the operation is on a 24-hour basis, the deck and rig lighting should be of a very high standard.

In general, all electrical apparatus, including telephones, within a certain distance from the rig must be of the explosion-proof type and, because of the wet conditions which often prevail, should be subjected to the highest standards of inspection and maintenance.

Lifeboats, lifebelts, and self-inflating dinghies are part of the normal equipment, but in order for these to be used efficiently should the occasion arise, it is essential that regular boat drills are made and a constant inspection of the equipment maintained.

Adequate fire-extinguishing equipment of all types must be carried, and considerable thought should be given to its disposition. There is no need to enlarge on the dangers of the fire hazard, but where this occurs on land there is a chance for the personnel to disperse, which is not so readily the case in offshore operations.

Over the past number of years much information on operating conditions has been collected, and from these safety codes have been compiled which deal with most probable eventualities. Notice boards indicating particular hazards are of considerable value, but there is no substitute for the most rigorous discipline

regarding safety precautions being enforced. This demands that only men of outstanding calibre should be placed in charge of these operations, because where respect and understanding exist the possibility of loss of life and serious damage is considerably reduced.

ACKNOWLEDGMENT

The author wishes to thank *Offshore*, of Conroe, Texas, for permission to reproduce four of the illustrations used in this section.

V. DRILLING

By W. P. Barker

With regard to the drilling operation itself, there is no fundamental difference between making hole on land and making hole from a platform or boat above a continental shelf; anything that can be done in a well onshore can be done in an offshore hole and, in general, methods of drilling and completion are the same in each case. Some modifications in the methods of spudding-in, however, have been found necessary, especially as the depths of the sea bed where the wells are drilled have become progressively deeper. The two marine problems, whether drilling in 10 ft of water in a swampy delta or in 500 ft or more depths in the open sea are:

(1) How to spud-in (*i.e.* commence drilling), and

(2) How to keep the sea out of the well.

In the majority of marine locations there is a considerable thickness of marine ooze on the sea bed which enables a conductor pipe, made up of 40-ft lengths welded together as it is run, to be driven into the ooze, sometimes as far as 150 ft. With the top of the pipe just underneath the rotary table, the conductor provides a waterproof lead-in for the drilling tools so that the well can spud-in and maintain circulation. As soon as a sufficiently good seat has been found in the rock under the ooze, surface casing is run and cemented. The conductor and the surface casing are then cut and secured together at floor level of the barge cellar, which allows blowout preventers to be installed, and a normal wellhead can be built up as future casing strings are run.

Where there is no layer of marine ooze above the sea bed, *i.e.* where the sea bed is hard rock, then some form of guide for spudding-in must be lowered from the platform and positioned under the rotary table at slack water on the sea bed. This is the position in the Persian Gulf, where ADMA operates, and ADMA solved the problem by incorporating a guide in, and lowering it with, the foundation framework for the future production platform for that particular well. This framework is a grillage and is lowered from the barge as soon as it has been raised on the well location.

As soon as the grillage with the guide is in position, the drilling tools are lowered into it, assisted by divers if necessary, and the well is drilled to a suitable depth for a conductor pipe (around 200 ft), the circulating fluid at this stage being sea water pumped down the drill pipe and precipitating the cuttings on the sea bed. The conductor pipe in ADMA wells is 30 inches diameter and is run in the same way that a welded casing string is run, and it is cemented very close to the bottom of the hole, *i.e.* cement is pumped into the

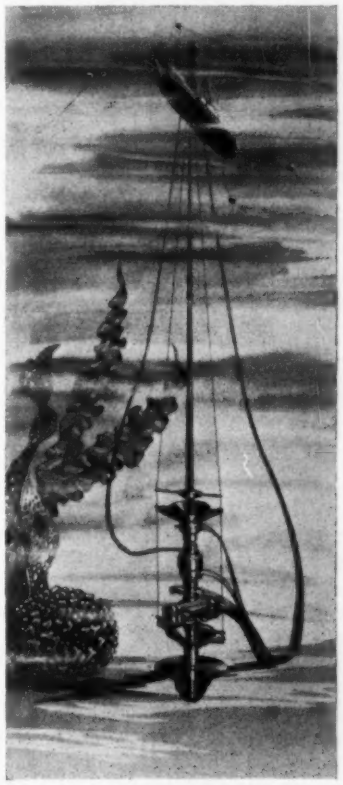


Fig 13

(Reproduced by courtesy of The Shaffer Tool Co., Brea, California)

conductor, where it naturally finds its own level inside and outside the pipe at sea bed depth. When the cement has set, the conductor provides a leakproof container which, after the cement inside has been drilled out, allows drilling with normal circulation to commence. A surface string of casing is run to about 500 ft, which, after normal cementation, is secured as

before to the conductor and allows an orthodox wellhead as on land to be progressively built up.

The above methods are satisfactory for wells drilled in up to 150 ft of sea water, *i.e.* for wells drilled from barges, from fixed platforms and tenders, or from self-elevating platforms. In water which is too deep for any sort of structure to rest on the sea bed, *i.e.* in depths down to 1500 ft, a system known as Sub Sea Drilling has been evolved in California (and to a lesser extent in Trinidad), where wells are drilled from an anchored surface vessel which is connected to the well on the sea bed by wire line guides only. All the normal wellhead fittings, such as blowout preventers, are located on the sea bed during the drilling of the well and remotely controlled from surface; in fact, the shut-off rams in one of the blowout preventers are used to seal the well while the tools are drawn up the wire line guides to the ship for bit change or for any other purpose. Normally a splined joint placed high up in the bit stand ensures a constant load on the drilling bit even if the surface vessel is under considerable movement, but should the sea become too rough for drilling the wire line guides are slipped from the ship and secured to buoys on the surface while the vessel rides out the storm elsewhere, resuming the drilling operation when the weather moderates. The deepest well to our knowledge drilled in this manner is one in the Gulf of Paria, in 85 ft of water, where 10,800 ft was reached.

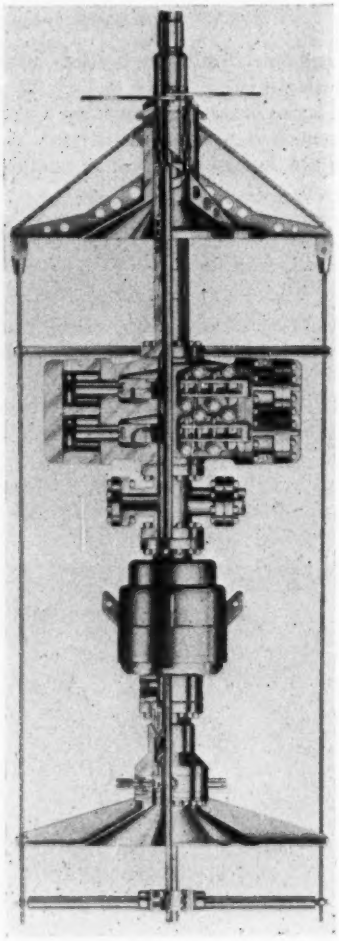


FIG 14

(Reproduced by courtesy of The Shaffer Tool Co., Brea, California)

The rig layout on most offshore rigs, whether diesel or diesel electric, is very similar to its counterpart on land except that as advantage can be taken of different deck levels, the outfit can be concentrated into a smaller overall area. This is a mixed blessing, as although considerable effort is saved working round the rig, if the well should start gassing, or gas-cut mud is being reconditioned, it is difficult to get out of a gassy area. Because of the gas hazard, owing first, to the restricted area available in an emergency, and secondly, to the heavier investment involved, casing programmes and blowout preventer equipment are somewhat more elaborate when marine drilling. Advantage is taken of the extra height available under the derrick floor to put in one or even two extra blowout preventers, in addition to which the preventer controls are duplicated, one set being on the derrick floor and the other some way away and out of the immediate vicinity of the well and shale shakers, where gas concentrations can be initially expected.

Besides the normal rig layout that has to be accommodated on an offshore platform or tender, it is now usual to install a Schlumberger or other electric wire line unit for logging and testing purposes. This unit, together with a laboratory for the geologist and petroleum engineer to work in, are fully equipped, and as such the platform is self-contained from a technical point of view.

Once the exploration well is started on its way with a wellhead installed, drilling proceeds exactly as on land to the completion of the well. In offshore areas where a number of rigs are drilling in comparative proximity, such as Lake Maracaibo or the Gulf of Mexico, the service companies co-operate as actively as they do ashore, only using boats, of course, instead of normal transport. If any particular testing or fishing tool or any special mud is required, or if acidizing or cementing services are necessary, a call to the service company is all that is required to bring round a vessel which can supply and run the requisite tool or pump the chemicals. The dry chemicals are blown up a 4-inch hose from the service vessel to the relevant storage silo or tank on the platform at approximately 60 tons/hr, while the fluids are generally measured and pumped up through a hose directly into the well itself.

On isolated locations, such as ADMA wells in the Persian Gulf, where service facilities do not exist, the operator himself must arrange to have every need anticipated, either on the platform or readily available at the base. It frequently happens that seas are too rough to unload bulk material for as long as 15 days at a time—when, in fact, the only method of transportation for either personnel, food, or very small items is by helicopter. Should drilling be held up during these periods because of waiting for bulk equipment such as casing or chemicals, owing to lack of foresight or to poor weather forecasting, a very expensive shut-down results.

Drilling mud is another problem. In this case, as the supply of sea water is unlimited, it is normally used as the base fluid for the drilling mud, and the mud storage facilities compare with a land-operated rig, *i.e.* some 8000 cu. ft. of reserve mud can be carried on the platform if required. Should severe lost circulation problems arise, and all stocks of mud be lost to the hole, it is always possible to keep the hole full by pumping sea water while remedial action is being prepared. All standard fluid equipment, such as a centrifuge or a degasser, together with a high pressure cementing/pumping unit, are normally installed, and some hundreds of tons of the chemicals necessary for mud fluid control are available on board; the more important chemicals, such as cement and barytes, are carried in convenient hoppers so as to be instantly available for use when required.

This considerable concentration of materials and men in a comparatively small space brings other problems. Although every effort is expended to isolate the living quarters at one end of the platform, it is quite impossible to damp out all the noise and vibration set up by the rig itself, besides such auxiliary equipment as the power generation, sea water pumps, and, in the summer, the air-conditioning units. These conditions highlight the problem of staff.

Normally there are on the platform or tender, at any one time, two complete shifts of some twenty-two men each, which include drillers, engineers, stewards, and cooks; these work alternate 12-hour shifts, while the third complete shift is at base or at home if this is near enough. Every three days one of these shifts is relieved by the third shift in rotation, so it will be seen that for a six-day period every man in a shift is working 12 hours a day and passing the other 12 hours in close promixity to working machinery, to his fellow men, and to possible danger. Apart, therefore, from the care required in choosing the type of man for the work, the quality of the supervision must be very carefully maintained. In addition to drilling the well, with its cementation and fishing problems, the supervisor has to organize the daily helicopter and boat services, watch the discipline at boat, fire, and blowout prevention drills, ensure that shift changes are punctual, and that no avoidable shut-down occurs in pushing tools and casing. In general, it may be said that staff and labour problems are more difficult to solve than any of the drilling problems involved in the operation.

VI. TESTING

By W. J. Baker

Testing follows the same general procedures as on land, and may be considered under the following two categories:

1. Drill-stem tests.
2. Completion tests.

Drill-stem tests are short exploratory tests of possible producing horizons, and may be carried out in open hole during the course of drilling, or in the cased hole by perforating the casing with bullets or shaped charges opposite the zone of interest. In either case it is necessary to isolate the zone to be tested from the rest of the hole by means of one or more packers set at the bottom of the drill pipe.

Completion tests are the final production tests of the well after it has been completed to produce from a selected zone or zones, and they are necessarily of longer duration than drill-stem tests.

The main problem in testing marine wells lies in the location of the testing gear and the disposal of any oil which may be produced. This difficulty is particularly acute in the case of completion tests of highly productive wells, where the volume of oil produced may be considerable.

Drill-stem tests do not necessarily always require the production of large quantities of well fluids to surface, and production may often be limited to the volume of the empty drill pipe; in such cases there are no problems. Similarly, if gas only is produced this can be vented through a vertical standpipe attached to the drilling mast, and water can be flowed into the sea. If, however, it is necessary to produce oil at surface, as in a completion test, a considerable amount of surface equipment and oil storage capacity have to be provided.

The surface equipment consists essentially of a gas–oil separator and flow tank with metering arrangements for both gas and oil. This is most conveniently mounted as a single unit on skids and set up on the drilling platform in any suitable position, such as on the pipe racks, and connected to the well by means of a flexible hose. The flow tank itself will in many cases provide sufficient oil storage capacity but, if not, additional tankage must be installed on the platform.

x 2

Alternatively, the oil may be flowed from the flow tank into a tank barge. The gas can be vented from the top of the drilling mast as previously described.

Another alternative is to install the surface equipment in a tank barge, but this has little merit unless there is no room for it on the drilling platform. Furthermore, it has the disadvantage of being at the mercy of the weather, which may severely limit its use in exposed locations. A test barge of this type has been used in BP's Persian Gulf operations and has proved quite satisfactory in fine weather.

It is common practice on land to dispose of oil and gas by flowing to a pit located at a safe distance from the well, where it is burnt. It is also possible to do this at sea by laying a flow line on the sea bottom and bringing it to surface at a suitable point, where a pile is driven to hold it in position. A saucer-shaped tray is fitted round the end section of the line out of reach of the waves in order to catch any oil which may drop from the flare.

It is recognized that there are certain hazards in testing, particularly in exploration wells, where the unexpected may always happen. While there is no greater likelihood of a mishap occurring at sea than on land, its results are likely to be far more disastrous and difficult to control. Testing procedures should therefore be planned with a view to reducing risks to a minimum and, of course, all possible safety precautions must be rigorously enforced while the test is being carried out.

VII. ECONOMICS

By C. N. Crosthwaite

It will be remembered that in the historical review of offshore exploration which prefaced this paper an aspect which was emphasized was its immensely greater cost compared with land operations.

In the preliminary phase of operations the relatively inexpensive methods of survey, such as aerial photography and surface mapping, are inapplicable. The main reliance has, therefore, to be placed on geophysics and the drill. An offshore seismic party operates off the Gulf Coast at an average cost of £25,000/month and averages 500 miles of shooting. This is about £50/linear mile or £12/station. A land party operates at a cost of £7150/month, averaging 50 miles of shooting, which amounts to £143/mile, or £36/station. In qualification of the above figures it should be mentioned that away from the Gulf of Mexico, and more especially in the remoter regions of the world, costs are considerably higher. Land work in North Africa might cost £500/mile, whilst offshore the comparable figure would be about £125.

These comparative costs show that, provided the weather is favourable, the greater speed possible with over-water operations enables a given area to be surveyed more rapidly and hence at a lower cost than an equivalent onshore area. On the other hand, offshore gravimeter surveys are considerably more costly than their onshore equivalents. Taking again average figures for operations in the Gulf of Mexico, a gravity crew will average 200 stations/month at a cost of £7140 or £36/station. On land, a party operates at a cost of about £3570/month and may be expected to average 600 stations—a cost of £6/station.

Another factor which adds to the cost of offshore exploration is the necessity for precise positional surveying out of sight of land. These surveys, which have to be made by methods specially developed for over-water work, may cost as much as £4300/month in out-of-the-way places. Where permanent directional aids have been established the chain service fee would be of the order of £1800/month.

It is in the next phase of exploration work, that of wildcat drilling, when costs become far higher than comparable onshore operations. One of the major operators in the Gulf of Mexico quoted the figure of $590,000 (£210,000) as the average cost of the offshore wells the company had drilled in 1957. This is approximately four times the cost of a similar well onshore. Let us consider the factors which account for offshore drilling being so much more costly.

First, there is the heavy capital investment involved. Where a big onshore rig might cost £180,000, its offshore counterpart would run from £250,000 to £290,000. Other major items additional to offshore drilling are the tender and platform. A tender will run somewhere around £540,000. The tender-type drilling platform costs £70,000 to £145,000 to build, including charges for the derrick and erection barges, while a self-contained platform costs about £360,000 to £540,000 and can cost up to £720,000. Large mobile platforms of the submersible or self-elevating type may cost between £1,430,000 and £1,790,000.

Another factor is higher labour costs. The labour force costs more both initially and during operations. Overtime hours are greater, and in addition costs are higher for housing, feeding, and transportation.

The problem of logistics offshore is considerably greater than in inshore operations. All phases of the operation are dependent upon an adequate onshore base and, particularly in remote regions of the world, establishment of essential base stores and other facilities is a very substantial capital expense. No less indispensable is the provision of adequate means of communication—by means of radar, wireless, and now often by helicopter—between the base and the offshore drilling units.

A further and far from negligible factor which

increases offshore operating costs is that of unfavourable weather conditions. It is frequently necessary to shut down operations for comparatively long periods to await the abatement of stormy weather. Just how hazardous the vagaries of tide and weather can be was exemplified in 1956 when Shell's heavy drilling platform, costing some £900,000, was wrecked in the Persian Gulf owing to a sudden storm.

The discovery of oil is by no means the end of the higher costs borne by the offshore operator. The capital expenditures required for the production of oil and its movement to onshore facilities are far higher than in similar land operations. Although in exposed areas subject to spells of bad weather barging is far from satisfactory, it is the first logical step when quantities are small. It is an expensive means of transport adding 40 cents/brl or more to production costs, depending primarily on the distances involved. The ultimate answer is a pipeline, if and when production warrants it, but offshore pipelines are so costly that only a major oil reserve can justify one. While total costs are at least double those onshore, this does not reflect the magnitude of increased construction costs. If pipe, which will cost about the same offshore as onshore, is excluded, the remaining investment will run from four to six times as great. A formula worked out by one company, using its own and other companies' offshore experience in the Gulf of Mexico, is $1/ft/inch of diameter plus $3/ft of linear length. These figures assume that construction is done during the most favourable season.

If costs in all phases of offshore operations are so much higher than onshore, the question arises why more and more companies are investing in offshore exploration both in the U.S. Continental Shelf and elsewhere in the world.

As is illustrated by the following figures, oil in the U.S.A. is becoming year by year increasingly harder and more costly to find.

The average drilling costs (plus leases, geological, geophysical, and general overhead costs) per well were $37,000 in 1939. By 1955 the average costs had risen to $107,000. The average footage drilled for new discoveries has more than doubled in the decade 1945–55. For every million barrels of new reserves 77,000 ft were drilling in 1945, in 1955 186,000 ft were drilled. Again the average reserves per successful well are diminishing and indeed not since 1953 has a major oilfield (100 million brl or over) been discovered. The fact is that most of the U.S.A. has been thoroughly explored, and as oil becomes harder to find the search is being turned towards more promising and relatively unexplored new, in some cases very remote, areas of the world. Whilst the comparison quoted earlier of the average cost of an offshore well in the Gulf of Mexico and a similar well onshore indicated the former to be four times more expensive, comparison with the cost of an onshore well drilled in a remote region of the world, such as Alaska or New Guinea, would present a very different picture. The cost of a wildcat in such a region might well exceed £1 million. Indeed, compared with onshore drilling in such regions, the high capital cost of mobile drilling barges has a number of compensating advantages. Offshore there are no locations to grade, no access roads to build—building a road through forest may cost as much as £25,000/mile—and when moving to a new location the time and expense of disassembly and re-assembly are saved.

There are so many variable factors to be taken into account in endeavouring to assess the relative costs of operating onshore and offshore in one section of the world, let alone in widely different parts, that a generalized conclusion as to the comparative costs would be as unwise as it would be misleading. The huge sums of money being spent in drilling both onshore and offshore illustrate the general guiding principle of oil exploration that it is a gamble demanding heavy stakes. As in other forms of gambling, the greater the stake the greater the possible reward.

DISCUSSION

E. E. Ferrin: These papers represent a complete but concise review of a very interesting subject, and I would like to compliment the authors on their work. They have traced the progress of world-wide submarine exploration for oil, but I would like to review briefly certain developments which have taken place in South Louisiana, where I was stationed during the period 1934–43.

In the oil industry it is essential to take advantage of the forces of nature rather than allow these factors to work against the operations. For instance, in certain sections of South Louisiana, particularly near the coast, the marshland is a difficult place to build suitable roads. Normal land-type seismic operations are quite costly because of the transportation problems involved, and this difficulty led to the use of marsh buggies and later to the employment of helicopters for the transportation of men and equipment. In some marshy areas board roads have been built to transport men and equipment to drilling locations, where the rig and all auxiliary equipment were supported by either wooden mats or wooden piling. The overall cost of this type of drilling operation was relatively expensive as compared to dry land operations, and as existing canals and bayous were usually near by, some operators were able to decrease their overall drilling costs by mounting their drilling equipment on barges and dredging canals to the drill site. By permanently installing drilling equipment on barges, and by utilizing the soft, marshy land for canals, the drilling operators were sometimes able to drill wells cheaper than by use of the former method.

During the onshore exploratory and development operations in South Louisiana it became evident that this vast oil region did not end at the present shoreline, and one enterprising operator set up a conventional rig on

piling in an area about one mile offshore from Cameron, Louisiana. This was by no means the first rig to be set up in open water, but this portion of the Gulf of Mexico is in an area where tropical hurricanes may strike at any time during the period of 15 July to 1 October. The original design of these offshore platforms constructed from many wooden pilings was such that they offered too much resistance to the wind and wave action. Every year, during the hurricane season, damage to the foundation and loss of drilling effort were experienced. This type of foundation was constructed by a conventional pile driver mounted on a barge, and there were many days when the water was too rough to operate this type of equipment. Thus the construction costs were extremely high.

In order to improve on the wood piling type of foundation, one of the construction contractors based at Harvey, Louisiana, started using a partially prefabricated drilling platform which was built onshore for a specific depth of water. These platforms constructed from tubular sections were actually templates through which piling of any required length could be driven for the required support. These prefabricated platforms could be installed at the location in a very short period of suitable weather. This type of foundation contained relatively few steel supports, which greatly reduced the resistance to wind and wave action and was more successful in resisting the tropical hurricanes than the former platforms constructed from wood piling.

This type of platform led to the development of large sea-going cranes, some of which had a lifting capacity of as much as 800 tons. By using these huge cranes on a 24-hour basis, complete unitized rigs, other unitized heavy sections of the platform, and complete bunk houses for 40 to 50 men could be moved in one piece, thus decreasing the amount of rigging-up time involved.

The authors have pointed out that other types of foundations for drilling equipment are used in the Louisiana Gulf Coast, particularly the self-contained, self-elevating, mobile type of offshore drilling outfits, but the basic unit for development operations involves some type of fixed platform. Mr Crosthwaite has indicated that some of these self-contained, self-elevating, mobile rigs may cost as much as £1,790,000. Offshore drilling operations conducted with this type of drilling outfit may have a daily cost ranging from £1800 to £4500. Thus it is extremely important that shut-down time be reduced to a minimum, and careful planning is essential.

The authors have not said too much about the series of mishaps to mobile drilling platforms used off the coast of Louisiana, and I believe it would be interesting to find out if they have any theories about the reasons why these huge drilling platforms should suddenly fail in service.

As an engineer, I cannot imagine anything worse than having a group of offshore oil wells on fire, as has recently been experienced by the CATC group in the West Delta area off the Louisiana coast, where seven wells, all of which were multiple producers drilled from the same platform, were involved in a major fire. It is normal procedure to use so-called "storm chokes" in all offshore wells, but it is a known fact that "storm chokes" do not operate very satisfactorily in a well either producing sand or at high rates of flow, and operating personnel sometimes remove these safety devices from the tubing string. I am wondering if the authors have any views on some method other than conventional "storm chokes" whereby such a disastrous fire could be prevented.

W. A. Roy: First of all I would like to thank Mr Ferrin for his most interesting contribution to the history of offshore drilling in the South Louisiana area over a period from 1934 to 1943, which is a long term in this young branch of our industry.

With regard to the series of mishaps which have occurred off the Louisiana coast, we are only aware of two major ones which have involved the total or partial loss of the structure. This does not mean that others have not occurred, but they must have been of a minor nature not to have been fully reported. In both these cases, namely, "Mr Gus" and the Trans-Gulf No. 10 Unit, the structure heeled over and soil failure was officially stated as being the cause in each case.

Where heeling over is experienced, some form of soil failure is the most likely cause, and this soil failure can be attributed either to scouring, in the case of soft sea bottom locations, or breakthrough, where the bottom is either hard or soft.

Considerable research work is at present being done on the prevention of scouring, and in most modern outfits anti-scour skirts are fitted.

In the case of breakthrough, pre-diving and pre-loading of the caissons in the self-elevating type of platform does help considerably in the elimination of this trouble.

As some members of the audience may not have encountered the terms "pre-diving" and "pre-loading" before, I will give a short explanation of what is meant by them. When the barge is floating, the caissons are lowered, on reaching the sea-bed they begin to penetrate, and as soon as they have reached a certain depth, which naturally depends on the nature of the sea-bed, the barge commences to rise. During this period maximum pressure is not required on the jacking pumps. When the barge is still in about 2 ft of water, *i.e.* when it still has some buoyancy, maximum pressure is put on the jacking pumps, and this has the effect of increasing the penetration. This operation is called pre-diving. As soon as the barge is clear of the water by about 2 ft, the pressure is momentarily taken off two diametrically opposed jacking pumps. This has the effect of very appreciably increasing the load on the other caissons, causing further penetration if the ultimate position has not been reached. This procedure is repeated on the other jacks, and by doing so a much greater load is imposed on the caissons than will be achieved during working conditions, *i.e.* the all-up weight of the barge and the maximum pull of the derrick which could be encountered from a stuck drill pipe. This operation is called pre-loading.

With regard to mishaps in general, my own feelings are that the number of these unfortunate happenings will decrease as time goes by and more and more experience is accumulated. The greater the appreciation of the hazards involved, the greater will be the pressure brought to bear on the problem, and this will ensure not only that design engineers and operating companies will try to solve the problems, but that they will enlist many representatives of other branches of science, such as oceanographers, experts on soil mechanics, and meteorologists, who have so much, in their own way, to offer to this new part of our industry.

W. J. Baker: The conventional type of storm choke, or safety valve, which Mr Ferrin mentioned as being rather unsatisfactory, works on the principle of having a restriction to flow in the form of a choke which sets up a differential pressure which, at a certain limiting rate of flow, shuts the valve against a spring, or in some cases against the weight of the valve. These storm chokes are usually installed at the bottom of the well, where they are out of the way and apt to get forgotten, and are not easily removed for inspection and maintenance. I think

that is probably why they sometimes fail to operate in an emergency. The present tendency seems to be to use a different principle. The valve is now hydraulically controlled from the surface and installed a few hundred feet below the well-head or, in the case of a marine well, below the sea-bed. It is fitted with a spring which tends to shut it, but at the same time it is kept open by a piston to which hydraulic pressure is applied from a surface control unit. The control unit can be adjusted so that it will shut the valve if the well-head flowing pressure increases or decreases beyond certain limits. The valve is very easy to test by simply releasing the hydraulic pressure, which will cause it to shut, and it can be opened again by applying pressure to the system. The conventional type is difficult to open once it has shut because it is necessary to equalize the well pressure across it. I must admit that I have had no experience with using this new type, but I think it is altogether more satisfactory than the old type, although it is more complicated. It does require a lot of extra equipment, including a special well-head and a small-bore hydraulic tube to convey the pressure down to the hydraulic mechanism.

Dr Milton B. Dobrin: I would like to ask a question relating to the overall economics. It seems to me that the present problem of the petroleum industry is over-supply, tariff barriers, low cost production in some areas and not in others, and so on. From the evidence we have been given it would appear that just every phase of the marine operation, of exploration, production, and distribution, costs much more than it does on land, and I have been wondering whether these marine areas are being considered in terms of long-term production prospects or whether the industry is thinking in terms of immediate use of such oil. In other words, are these marine reserves something that may have to be set aside for exploitation in five years, ten years, fifty years, or what? Perhaps my thinking is all wrong and that in actual fact a lot of this oil which is being found in the marine areas is to be exploited immediately.

C. N. Crosthwaite: Although, as Dr Dobrin has said, over-supply is one of the present problems of the petroleum industry, the growth of oil consumption has only been temporarily retarded, and indeed it is anticipated that world demand will double in ten years' time. The costly search for new reserves in marine areas therefore betokens the industry's confidence in a continuing expansion of oil consumption. How soon marine discoveries will be exploited will depend on such factors as the location of the discovery in relation to markets, and the type of oil. As shown in Fig 3 in the papers, production of oil and gas is already being rapidly developed from the new Louisiana offshore fields, and were it not for the drastic cut in allowables, development would have been substantially quicker. But generally speaking to-day's exploration, whether of land or marine areas, is an investment for the medium or long term—for exploitation in, say, five to twenty years' time.

Dr D. T. Germain-Jones: It is stated in the geophysical section of the papers, and mentioned also by Mr Jackson, that depth and bottom conditions may cause such channelling of energy that all reflections are lost. How do we overcome that one? If it happens all over the area, what does the geophysicist do next?

A. B. Malone: This phenomenon, which was known, incorrectly in my opinion, as "wave-guide," has been under consideration recently. It was supposed that all the energy from the shots was caught in an energy trap—a non-attenuating medium bounded by good reflecting surfaces at the top and bottom of the water layer. It was further supposed that none of the energy penetrated into the underlying strata. Generally speaking, there have been one or two recent papers in which the authors suggest that this is not always the case, and that this "singing" or reverberation that does undoubtedly occur is sometimes caused by some of the energy being reflected back from deeper beds, which sets the surface layers back into vibration again. One of our contractors believes that by passing the energy recorded through an inverse filter, he can remove the oscillation and bring out the energy coming from the deeper beds. Some remarkable improvements in the record sections have been produced in this way.

Dr D. T. Donovan: I thought the picture of the part played by geologists, as presented in this symposium, unduly pessimistic. I may say that I am a geologist, of course. While it is difficult to see how one could do any geological work in areas which are covered by marine ooze or recent sedimentary cover, where there is no such cover there are several very considerable advantages. The first is that the rock floor may be almost continuously exposed, which very seldom happens on land. The relief is usually very low, one can almost consider it as a plane surface, so that the interpretation of the outcrop pattern is easy. Also, the considerations that make for ease of seismic survey, which were mentioned in the case of geophysics, namely, steaming along parallel courses and so on, apply equally well to geology. My own slight experience in submarine geological survey does indicate that, at least in certain cases, with recent developments in acoustic survey, one can actually obtain more detail in mapping the sea-bed than in mapping land.

H. Hyams: I am not a geologist, a production engineer, in any way connected with exploration and drilling, nor am I even an economist, so the question I am about to put will not, I hope, be regarded as too nonsensical, because even my mathematics or arithmetic in the question may be completely wrong.

Mr Elder states that in 1947 Pratt calculated that the potential oil reserves of the Shelf and the Slope would be about one million million barrels, which Mr Elder says is about three times the amount of production plus proven reserves so far found on land. If the Shelf alone is taken, the proven reserves figure would, we are told, be something of the order of 150 to 200 thousand million barrels. If, then, as the author indicates, we allow for 25 to 30 per cent reduction in that figure for areas bordering the Shelf, which would probably be unprospected land, the Shelf reserves will be reduced to somewhere between 100 to 150 thousand million barrels. It will be noticed that I have reduced the million million barrels now to somewhere around 150 thousand million.

The figure that I have for 1957 is that the land proven oil reserves at that time amounted to 230 thousand million barrels, so that if this figure is correct, and even based on Pratt's calculations, the proven oil reserves on land are at least as great as those calculated to exist below the great oil lakes and the sea itself.

Mr Elder is probably correct in saying that the reserves of the Shelf are possibly of a high order, but what I would ask him is: Would he not agree that it is possibly unwise to permit people to believe, on the relatively scanty data available at this early stage, that we have the best part of one million million barrels of proven oil reserves lying below certain lakes and the Continental Shelves and Slopes? In other words, I am wondering whether we

are not possibly over-estimating the future possibilities of production from beneath the Shelf and the lakes, in relation to what are still very large possibilities on land itself.

S. E. Elder: So little is known, relatively and geologically speaking, of the vast area of the Continental Shelf that estimates are really a matter of individual opinion equally difficult to prove or disprove. My opinion, as implied in the paper, is that I consider some of the estimates to be high, and in this respect I agree with the chairman's remarks.

R. H. W. Hamilton: I would like to ask the panel whether they feel that the much publicized project of drilling a well to a great depth into the crust of the earth, and starting in a considerable depth of water, is in fact a practical proposition for this generation?

W. P. Barker: I answer this question in the affirmative and consider this well will be drilled within the next fifteen years.

It will be a difficult drilling problem and, as such, very expensive. As this is a scientific research project rather than a potential oil-well, it will, no doubt, be sponsored by and paid for from government or even international sources.

The meeting then closed with a unanimous vote of thanks to Mr Jackson and the authors of the papers.

CONTRIBUTED DISCUSSION

B. O. Ashford: I have been reflecting on some of the practical implications involved in a vast extension of offshore drilling activities, particularly the matter of the marine corrosion which could occur in such complex steel structures as drilling barges.

The particular point I had in mind was the sea water corrosion of the intermittently submerged portions of the barge and the supporting legs, which must include a large area of mechanically working parts which could not readily be dealt with by protective coatings.

W. A. Roy: Mr Ashford's appreciation of the considerable corrosion problem which is encountered in all offshore operations is a very pertinent one, and is one which is ever present in the minds of all operating companies. In the case of the self-elevating type of barge the submerged portion of the caissons can be adequately protected by cathodic means but, as he rightly says, it is not easy to apply some form of protective covering to the splash zone of the caissons because this would be stripped off during the elevating or lowering of the barge. Scraping and painting can, however, be done in the splash zone area when fine weather permits. The general practice with regard to the caissons is to include a substantial increase in their thickness as a corrosion allowance and to take the barge out of service at periods, which vary with the different operating companies' practice, for servicing and making good of any corrosion damage.

In the case of fixed-height platforms it is, of course, possible to apply protective coatings in the splash zone areas, and for this purpose some of the more newly developed epoxy resins are used. In a number of cases operating companies have found it worthwhile to sheath this area with monel metal.

With regard to installed equipment and the hull and deckhouse paintwork, this can, to a large extent, be looked after during service, but to do so it is necessary to introduce a rigorous maintenance procedure. The work must be kept going all the time and constant inspection and instant remedial action taken, otherwise serious trouble will quickly be experienced.

COLOUR OF REFINED PETROLEUM OIL BY THE SAYBOLT CHROMOMETER

PROPOSED WITHDRAWAL OF IP 18/53

STANDARDIZATION COMMITTEE have recently approved a recommendation by the Colour Panel that method IP 18/53, Colour of Refined Petroleum Oil by the Saybolt Chromometer, should be discontinued as an IP Standard Method. However, because this method has been widely used for many years and appears in several specifications, it has been decided that this recommendation should not be put into effect for one year, during which any objections may be lodged for consideration.

The reasons for the Colour Panel's recommendation are as follows:

(1) The hue of the Saybolt glasses is different from that of many present-day white products. This makes it impossible to obtain a proper match and the precision of the method is thereby greatly impaired;

(2) The instrument is essentially of American manufacture and difficulty has been experienced in obtaining components in Britain. Recent modifications have in fact rendered obsolete most of the instruments now in use;

(3) A satisfactory alternative already exists in the IP Lovibond instrument, IP 17/52.

All objections or other observations regarding the proposed withdrawal of the Saybolt method should be sent as soon as possible to: The Technical Secretary, The Institute of Petroleum, 61 New Cavendish Street, London, W.1.

THE POTENTIALITIES OF COATED CAPILLARY COLUMNS FOR GAS CHROMATOGRAPHY IN THE PETROLEUM INDUSTRY *

By D. H. DESTY,† A. GOLDUP,† and B. H. F. WHYMAN †

SUMMARY

The application in the petroleum industry of the coated capillary columns recently described by Golay and Dijkstra has been examined. In conjunction with the new sensitive ionization detectors they enable very high separation efficiencies to be attained and offer the possibility of much reduced time of analysis.

A complete apparatus operable up to 250° C incorporating a flame ionization detector and a novel sample introduction system for both gaseous and liquid samples has been developed. Results obtained with a variety of petroleum products from gases to high-boiling liquids are described and used to illustrate the potential uses of these columns in the petroleum industry.

Some considerations have been given to the theory of coated capillary columns and the Golay and van Deemter equations for the height equivalent to a theoretical plate are compared.

NOMENCLATURE

n	Total number of theoretical plates
H	Height equivalent to a theoretical plate (HETP)
H°_{min}	Minimum HETP when resistance to mass transfer in the liquid phase is absent
u	Average linear gas velocity
λ	Correction factor for packing irregularities
γ	Correction factor for tortuosity of gas channels
d_p	Average particle diameter
D_g	Coefficient of gaseous diffusion
D_l	Coefficient of liquid diffusion
k'	Ratio of fixed phase to moving phase capacity
d_f	Average liquid film thickness
r	Average radius of capillary tube
F_l	Volume fraction of liquid
F_g	Volume fraction of gas
k	Partition coefficient

INTRODUCTION

THE possible use of micro columns, a few tenths of a millimetre in diameter, in gas chromatography was first mentioned by A. J. P. Martin as early as 1956.[1] The first experimental work on capillary columns coated with a thin film of stationary phase was, however, described by Golay in the autumn of 1957 [2] while discussing the theory of such columns. In presenting a later paper [3] extending this theory, the same author produced two chromatograms demonstrating very effectively the ease with which high column efficiencies could be attained. These results aroused considerable interest in this new technique and stimulated the present authors to investigate the possibilities of these new columns for examining the very complex mixtures occurring in petroleum, where high separation efficiency would be particularly valuable in separating isomeric hydrocarbons.

EXPERIMENTAL

Although a detector of the argon ionization type specially designed for use with capillary columns has recently been described,[4] the authors have successfully employed a simple form of the flame ionization detector developed by McWilliam.[5] No experimental apparatus for use with these columns has so far been published, and it seems worthwhile at this stage to give some details of equipment which has been employed.

Apparatus

The general arrangement is shown in Fig 1. The column, detector, and sample introduction system are attached to a simple thermostat heated electrically and controlled by a bimetallic regulator. Nitrogen is normally used as carrier gas and enters the apparatus from a constant pressure source through the sample introduction system passing through the column at flow-rates generally less than 1 ml/min. The exit gas is mixed at the column outlet with a faster stream of hydrogen (*ca* 20 ml/min), fed again from a constant pressure source, through a fixed restriction, and the mixed stream burnt at the jet of the detector in a current of dust-free air. The ionization current passing through the flame under the influence of the polarizing potential applied to the collector is fed to a commercial vibrating reed electrometer (Electronic Instruments Ltd " Vibron " Model 33B) which acts as an impedance changer between the detector and potentiometric recorder.

Thermostat

The thermostat chamber (Fig 2) takes the form of a thick walled aluminium pot fitted with a thick aluminium lid, on which is mounted the column, detector, sample introduction device, and the gas flow systems. In this way all critical parts of the apparatus are maintained at or near the operating

* MS received 7 May 1959. Presented at an informal symposium organized by Gas Chromatography Discussion Group (Hydrocarbon Research Group, IP) at Imperial College, London, 10 April 1959.

† The British Petroleum Co. Ltd.

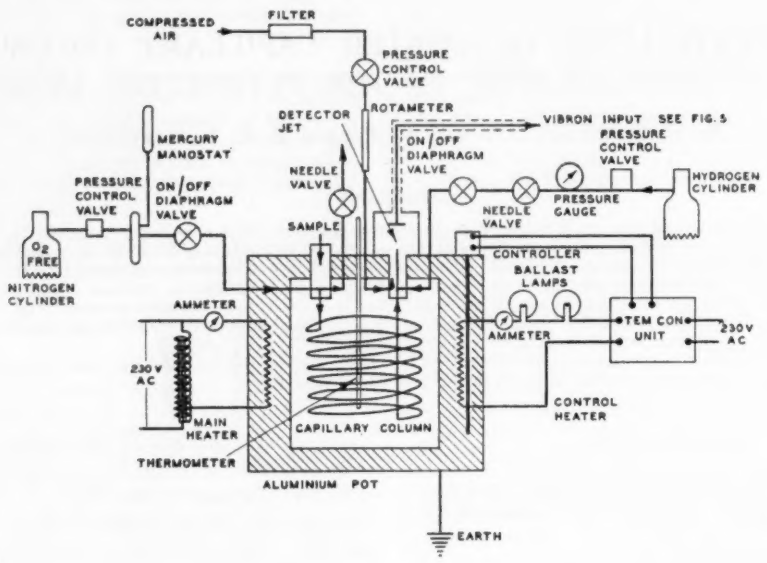


FIG 1

GENERAL ARRANGEMENT

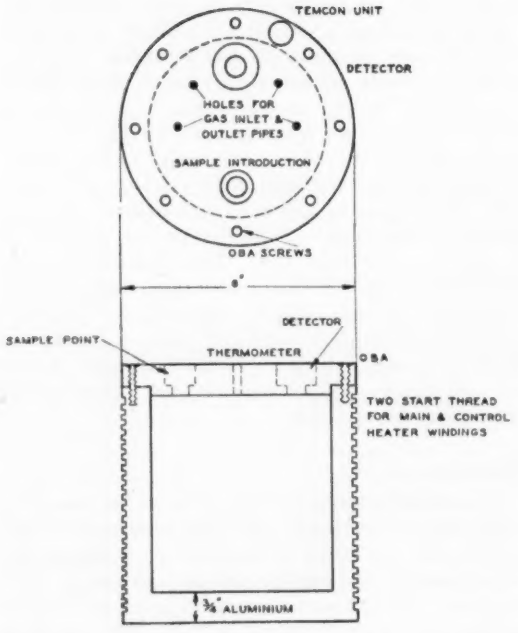


FIG 2

THERMOSTAT CHAMBER

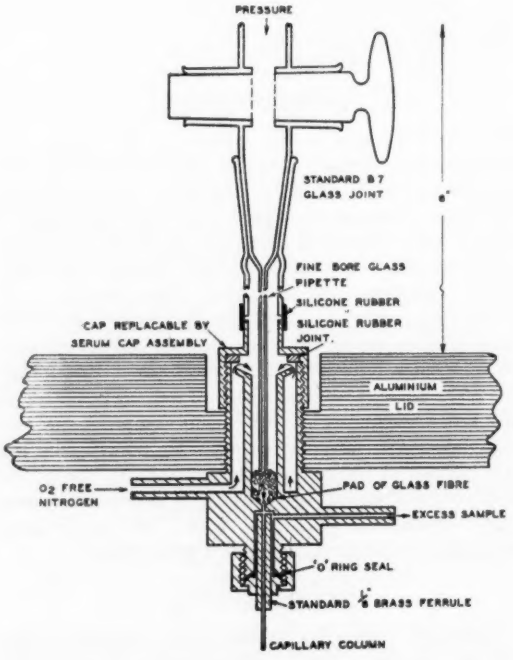


FIG 3

SAMPLE INTRODUCTION SYSTEM

temperature and the various gas streams entering it are preheated. In addition, condensation between column and detector is prevented and the arrangement is suitable for operation at high temperature (above 200° C).

Sample Introduction Device

The importance of introducing a sharp plug of sample of small enough size has been stressed by many authors in connexion with conventional packed columns. Keulemans [6] has shown that the maximum sample size above which peak broadening occurs is directly proportional to the product of the plate capacity and the square root of the total number of plates (n). In addition, this small sample, as discussed by van Deemter,[7] must be introduced on to a proportion of the total plates which falls rapidly as the column efficiency increases (according to the reciprocal of the square root of n). It will be appreciated, therefore, that with capillary columns, where plate capacities are of the order of a thousand times lower than packed columns and efficiencies ten or more times higher, the introduction of extremely small samples very sharply becomes of paramount importance.

The authors have attempted to achieve this by the device shown in Fig 3, which may be fitted with a rubber septum for the injection of vapour samples or the attachment shown for liquids. The inlet nitrogen stream, after passing through a preheater, enters the device and undergoes a dynamic division over the end of the capillary column, by far the largest proportion escaping through the external adjustable needle valve. In this way only a very small fraction (<5 per cent) of a vapour sample introduced before the division point through the rubber septum from a syringe passes into the column. Moreover, since the initial pellet moves very rapidly over the end of the capillary this small fraction enters as a compact plug. With liquid samples the septum is replaced by the micro-assembly shown in Fig 3. The pipette is loaded externally and then attached at the ground glass joint so that the sample contained in the narrow glass capillary is held in a comparatively cool region in the glass tube above the entry point. It is ejected by slight air pressure and falls on to a short wad of glass fibre placed as shown.

It has been found with both forms of the device that division ratios greater than 20 : 1 are necessary to avoid peak broadening, particularly with fast-moving components. With liquid samples, where 20 to 100 μg are actually introduced and 0·1 to 2 μg enters the column, satisfactory vaporization of the sample takes place with the glass fibre held at the operating temperature. No evidence of fractionation in the division has been observed, but this point has not yet been checked exhaustively over a wide range of division ratios and sample sizes.

Column Mounting and Coating

A variety of capillary tubing has been used to construct columns, but none has had an outside diameter greater than 0·125 inch. In order to use a fixed size of connexion, therefore, all the columns have been fitted with short sleeves about 1 inch long and 0·125 inch diameter, with a suitable solder or cement, and these are held in simple O-ring type compression seals at the inlet and outlet. This has the advantage that columns are easily interchangeable and dead volume in the connexions is reduced to a minimum.

The coating procedure described by Golay [2, 3] is attractive in that, as the film is deposited at a virtually static liquid–gas interface, good uniformity is to be expected and determination of the total quantity of stationary phase in the column is easy. It has the serious disadvantage, however, that coiling of the tube on a suitable former can be carried out only after or during the coating operation. The dynamic method described by Dijkstra [8] has therefore been adopted, in which a quantity of a solution of the stationary phase in a light solvent is passed through the tube and residual solvent removed from the thick solution film is left behind, by a slow stream of nitrogen, sometimes at slightly elevated temperatures. It is important to move the solution through the tube at slow velocities (<10 cm/sec) and to avoid a sudden change in velocity as the last of the solution leaves the column. To this end, the minimum quantity of solution necessary to eject a small liquid pellet from the column is used as this minimizes the pressure differential across the column and also the change in flow as the last of the liquid leaves.

With thin walled capillary it is possible to determine the bore of the capillary and the total quantity of stationary phase in a prepared column gravimetrically. The film thickness may then be calculated. Table I gives results for a gold and a copper capillary coated with squalane in n-pentane under the conditions given above. The data, which are less precise for the copper tube as it has a thicker wall, are, of course, average values over the whole

TABLE I

Data on Gold and Copper Capillaries Coated with Squalane (from Solution in n-Pentane)

	Gold capillary	Copper capillary
Weight of column (g)	51·6600	106·8004
Volume of bore (ml at 20° C)	0·505	0·762
Length (cm)	1497	1220
Average bore diameter (μ)	207 ± 1	282 ± 3
Concentration coating solution (% v/v)	10	20
Weight of squalane (mg)	9·2 ± 0·1	15·9 ± 0·2
Average film thickness (μ)	1·17 ± 0·05	1·8 ± 0·1
External diameter (mm)	0·51	1·1

length, and it is hoped to develop methods of studying the uniformity both of the bore and of the film thickness along the length.

Hydrogen Injection System and Detector

The construction of the hydrogen injection system at the end of the column is shown in Fig 4. The

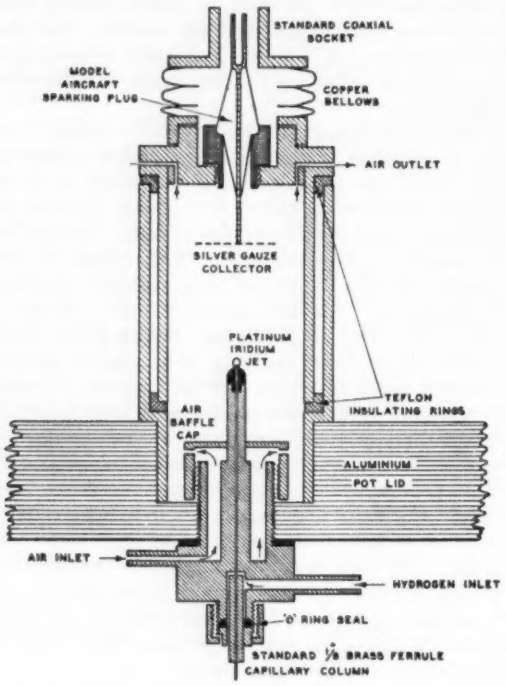


FIG 4

COLUMN OUTLET AND DETECTOR

outlet nitrogen stream from the column is mixed at the end of the capillary with a faster stream of hydrogen emerging from around the top of the sleeve fixed at its end. Thus all dead spaces are avoided. The mixed stream then passes up the jet and burns at the platinum/iridium tip, which has a hole about 0·012 inch through it. The air necessary for combustion is fed into the jet unit below the thermostat lid and flows up around the jet, issuing through a baffle cap at the bottom of the flame chamber. Air flow rates in the range 200 to 500 ml/min are normally employed.

The flame chamber is formed by two concentric cylinders of brass, separated by insulating Teflon rings at the top and bottom, and sits in a shallow well in the thermostat lid. The collector, in the form of a disk of silver gauze about 1 cm in diameter, is supported by a short extension from the centre electrode of a miniature sparking plug held in the chamber cap. The distance between the collector and jet is about 1·5 cm. The air leaves the chamber through a series of small holes and grooves machined in the top cap. To provide shielding for the high impedance connexion to the top of the sparking plug, a short length of copper bellows is mounted on the top of the chamber cap and this supports a standard coaxial socket. This construction allows the input coaxial screen, chamber top cap, and the inside of the chamber to be held at " guard " potential, thus minimizing leakage. In addition, the coaxial socket remains sufficiently cool, even when operating at 250° C, to use polythene insulated anti-microphonic coaxial input cable.

The input circuit used with the detector is shown in Fig 5. The sensitivity is not significantly dependent on the polarizing potential, and we now use a 60 V HT battery in the low impedance lead with the input circuit floating above earth. The jet is, of course, earthed and the collector normally negative. The standing current with no sample is usually in the range 10^{-12} to 10^{-11} amp, and with an input resistor of 10^{-10} ohm and the Vibron sensitivity at 100 mV, some backing off is often required. This is provided by applying to the input resistor a small current from a grid bias battery in series with a 10^{12}-ohm resistor, in the opposite sense to the current from the detector. A potentiometer across one cell of the battery gives fine control, and coarse adjustment can be made by moving the potentiometer along the battery. In this

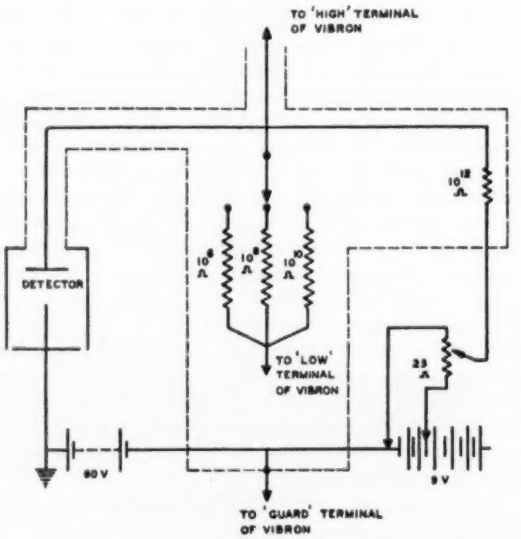


FIG 5

INPUT CIRCUIT OF VIBRON ELECTROMETER

way a range of backing off currents from nil to 10^{-11} amp can be provided.

This arrangement may be easily adapted for direct integration of the ionization current from the detector by substituting a polystyrene condenser of appropriate value for the input resistor.

It is difficult to express the sensitivity of the detector because of the dilution which occurs at the end of the column, particularly since it appears substantially independent, within reasonable limits, of the hydrogen flow rate. Based on the nitrogen carrier gas, however, the noise level represents a concentration of about 10^{-7} μg/ml for *n*-butane at room temperature.

Column Materials

Commercially available metal capillary columns of approximately 0·01 inch bore have been used for most of the experimental work and are suitable for the examination of hydrocarbon blends normally encountered in the petroleum industry. Both stainless steel and copper have been used successfully at operating temperatures up to 250° C, although, with the latter metal in particular, rapid decomposition of the stationary phase (Apiezon L grease) occurred above 200° if air was accidentally admitted into the column. Gold has also been used, but is obviously too expensive for routine use. Recently,[9] an apparatus has been developed for drawing glass capillary columns, and although the potentialities of these have not yet been fully explored, preliminary results indicate that they have considerable promise.

In the light of the work on metal helices[10,11] and silver-coated firebrick,[12] it would be expected that the absence of the inert support in capillary columns would enable polar solutes to be examined with a non-polar stationary phase without giving rise to tailed peaks such as are often encountered with conventional packed columns.[11] Our results do not confirm this view; indeed, we have found that, with stainless steel in particular, polar materials such as ethyl alcohol, diethyl sulphide, acetone, ethyl acetate, all give rise to tailed peaks when squalane is used as the stationary phase. Similar results are found with copper and gold, although the extent of adsorption is considerably reduced with these metals. On stainless steel, in the absence of any stationary phase, even non-polar or weakly polar solutes are retained, and it is possible to separate simple hydrocarbon blends. Presaturation of the carrier gas with water vapour[13,14] considerably reduces both the degree of asymmetry of the peaks and the retention time. As is usual with adsorption effects, the latter is also dependent upon the sample size, being reduced as the sample size is increased.

Uncoated glass capillaries, as would be expected from the polar nature of the wall, give rise to considerably skewed peaks with polar solutes, but this is markedly reduced when the stationary phase is applied. The reason for this difference from metal capillaries is at the moment obscure, but is probably the result of a more uniform film being formed over the very smooth wall of the glass capillary. Under the microscope there is a very considerable contrast between the smoothness of the glass and metal capillaries.

TABLE II

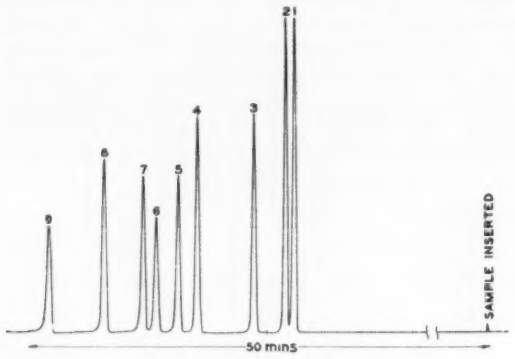
Capillary Column Data

Column Description:	1 *	2 *	3	4	5	6	7	8
Tube	Stainless steel	Stainless steel	Stainless steel	Copper	Copper	Stainless steel	Glass	Glass
Length (m)	45·7	45·7	48·5	82	82	15·5	36·5	36·5
Bore diameter (cm)	0·025	0·025	0·028	0·028	0·028	0·025	0·015	0·015
Stationary phase	Didecyl-phthalate	Didecyl-phthalate	Squalane	Squalane	Squalane	Apiezon L	Squalane	Squalane
Sample:								
Sample	C_6 Hydro-carbons	C_8 Aromatics	C_4–C_6 Hydro-carbons	C_7 Paraffins	C_8 Paraffins	n-C_{13}–C_{20} Paraffins	Reformate	C_4–C_6 Hydro-carbons
Approx sample size (μg)	—	—	2	2	2	0·5	0·02	0·01
Approx No. of components	6	4	9	9	17	7	25	9
Operating Conditions:								
Eluent gas	Helium	Helium	Nitrogen	Nitrogen	Nitrogen	Nitrogen	Nitrogen	Nitrogen
Inlet pressure (lb/in²)	20	10	5	15	15	1·7	10	7
Flow rate (ml/min)	0·96	0·48	0·40	0·60	0·60	0·50	0·25	0·20
Linear gas velocity (cm/sec)	35·7	15·9	8·5	11·9	11·9	8·6	8·3	7·2
Temperature (°C)	60	70	20	72	72	245	42	20
Performance Data:								
Component	*n*-Hexane	*o*-Xylene	*n*-Hexane	2-Methyl-hexane	3-Ethyl-heptane	*n*-Nona-decane	*n*-Heptane	*n*-Hexane
Efficiency	13,000	44,000	28,000	106,000	117,000	25,000	60,000	100,000
HETP (mm)	3·5	1·0	1·8	0·77	0·70	0·62	0·61	0·37
HETP/r	28	8·0	13	5·5	5·0	5·0	8·1	4·9
Resolution	36	90	64	120	125	60	98	94
k'	2·6	13·5	6·3	3·3	6·8	3·8	2·2	2·1

* Golay's results.[3]

Data on some of the columns we have examined are given in Table II, together with Golay's results.[3]

Our first experiments were carried out with thick-walled stainless steel capillary, with an outer diameter of 0·125 inch (Table II, Column 3). Results were encouraging, but the tubing was cumbersome, heavy, and, in addition, expensive. Thin-walled capillary



CHROMATOGRAM 1

Separation of the C_7 Paraffins

Column:	250 ft 10 thou id copper
Stationary phase:	Squalane
Temperature:	72° C
Carrier gas:	Nitrogen
Inlet pressure:	15 psi
Flow rate:	0·6 ml/min
Sample size:	Approx 2 μg
Efficiency:	106,000 theoretical plates (3-ethylpentane)
Detector:	Flame ionization

Components

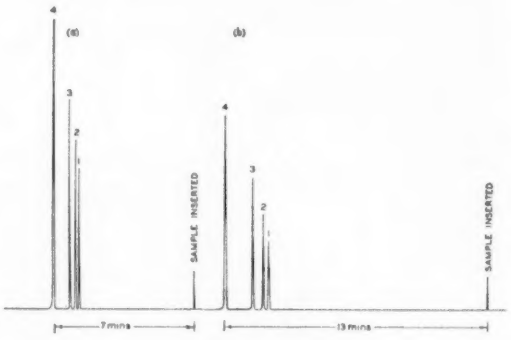
1. 2,2 Dimethylpentane
2. 2,4-Dimethylpentane
3. 2,2,3-Trimethylbutane
4. 3,3-Dimethylpentane
5. 2-Methylhexane
6. 2,3-Dimethylpentane
7. 3-Methylhexane
8. 3-Ethylpentane
9. *n*-Heptane

is obviously much more attractive for the purpose, and copper tube of about 0·045 inch outer diameter and the same bore was obtained. A 250-ft length can be compactly coiled on to a metal spool only 3 inches high and about 2 inches across. A column of this type (Column 4) coated with squalane (from a 20 per cent v/v solution in *n*-pentane) gave a complete separation of the nine C_7 paraffin isomers (Chromatogram 1) at 72° C and the HETP compared well with that obtained by Golay [3] (Column 2). The highest efficiency so far measured has been obtained under the same conditions (Column 5) with the slower moving C_8 paraffins (*ca* 120,000 plates), 14 maxima being observed with a mixture containing 17 of the possible 18 isomers. This column has also been employed to demonstrate the rapidity with which analyses may be carried out. In Chromatogram 2 the separation of methane, ethane, propane, and *n*-butane is shown, operating at 80° C, in which the four components emerge from the column in about 1 minute and the total analysis time is only 7 minutes.

The efficiency of the columns seems to be maintained at high operating temperatures above 200° C. A 50-ft thin-walled stainless steel column (Column 6) coated with Apiezon grease L, at 245° C produced 25,000 plates with a *n*-paraffinic mixture and the HETP (0·62 mm) was similar to that obtained at temperatures below 100° C with copper tube.

The development of a method of drawing glass capillaries enabled columns of somewhat smaller diameter to be examined and, as already pointed out, the bore of these glass capillaries being much smoother than the commercial metal tubes employed, it seems likely that the film of stationary phase is more uniform. At room temperature a 120-ft column (Column 8) having a 0·006 inch bore coated with squalane gave 100,000 theoretical plates and an HETP of 0·37 mm, the lowest so far obtained (Chromatogram 3). This same column (Column 7) at 42° C had a somewhat higher HETP, but accomplished as good a separation of the C_7 and C_8 paraffins as the 250-ft copper columns, but in about half the time.

Preliminary work on the determination of retention volume data has been carried out with the aim of



CHROMATOGRAM 2

Rapid Separation of Methane, Ethane, Propane, and n-Butane

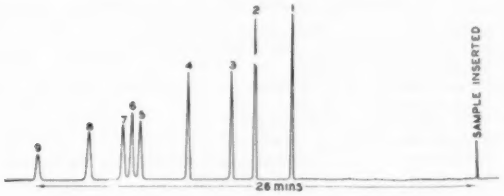
Column:	250 ft 10 thou id copper
Stationary phase:	Squalane
Temperature:	80° C
Carrier gas:	Nitrogen
Inlet pressure:	(*a*) 25 psi
	(*b*) 15 psi
Flow rate:	(*a*) 1·0 ml/min
	(*b*) 0·5 ml/min
Sample size:	0·01 μl
Efficiency:	150,000 theoretical plates
Detection:	Flame ionization

Components

1. Methane
2. Ethane
3. Propane
4. *n*-Butane

comparing the values obtained with those determined with packed columns under similar conditions. The work, however, is handicapped by the difficulty of

measuring accurately the position of the "air-peak," since the detector responds only to organic materials.



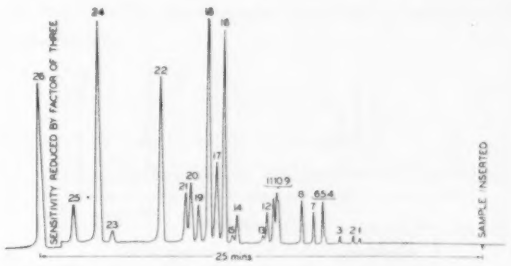
CHROMATOGRAM 3

Separation of a C_4–C_6 Hydrocarbon Blend

Column:	120 ft 6 thou id glass
Stationary phase:	Squalane
Temperature:	20° C
Carrier gas:	Nitrogen
Inlet pressure:	7 psi
Flow rate:	0·20 ml/min
Sample size:	Approx 0·01 μg
Efficiency:	100,000 theoretical plates (*n*-Hexane)
Detector:	Flame ionization

Components

1. *n*-Butane
2. *iso*Pentane
3. *n*-Pentane
4. 2,2-Dimethylbutane
5. *cyclo*Pentane
6. 2,3-Dimethylbutane
7. 2-Methylpentane
8. 3-Methylpentane
9. *n*-Hexane



CHROMATOGRAM 4

Product from Reforming of Methylcyclohexane

Column:	120 ft 6 thou id glass
Stationary phase:	Squalane
Temperature:	42° C
Carrier gas:	Nitrogen
Inlet pressure:	10 psi
Flow rate:	0·25 ml/min
Sample size:	0·02 μg
Efficiency:	60,000 theoretical plates (*n*-Heptane)
Detector:	Flame ionization

Components

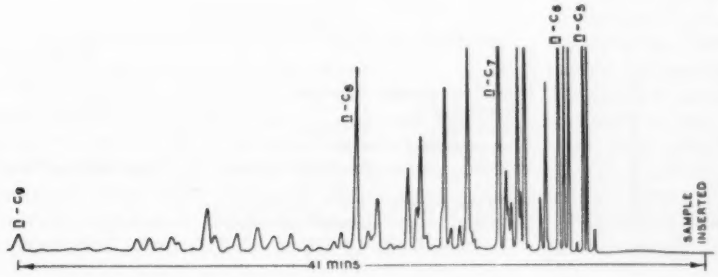
1. *iso*Pentane
2. *n*-Pentane
3. 2,2-Dimethylbutane
4. *cyclo*Pentane
5. 2,3-Dimethylbutane
6. 2-Methylpentane
7. 3-Methylpentane
8. *n*-Hexane
9. Methyl*cyclo*pentane
10. 2,2-Dimethylpentane
11. 2,4-Dimethylpentane
12. Benzene
13. 2,2,3-Trimethylbutane
14. 3,3-Dimethylpentane
15. *cyclo*Hexane
16. 2-Methylhexane
17. 2,3-Dimethylpentane
18. 3-Methylhexane
19. *trans*-1,3-Dimethyl*cyclo*pentane
20. 3-Ethylpentane *cis*-1,3-Dimethyl*cyclo*pentane
21. *trans*-1,2-Dimethyl*cyclo*pentane
22. *n*-Heptane
23. *cis*-1,2-Dimethyl*cyclo*pentane
24. Methyl*cyclo*hexane
25. Ethyl*cyclo*pentane
26. Toluene

APPLICATIONS

Little attention has been paid to the application of these columns to the separation of hydrocarbon gas mixtures, as, in general, there is little need for high separation efficiency in the analysis of samples normally encountered. The possibilities of the technique for rapid monitoring of process streams should not, however, be ignored.

It is in the separation of mixtures containing isomeric hydrocarbons of similar structure that the columns have the greatest potentialities. This has already been illustrated in the results with blends of the C_7 and C_8 paraffins, and another example is shown in Chromatogram 4, where a product obtained by catalytic reforming of methyl*cyclo*hexane has been examined. Over 20 components have been separated and identified in as little as 25 minutes. With straight-run gasolines boiling up to 150° C the total number of peak maxima observed with a capillary column having an efficiency of approximately 60,000 plates is about three times that obtained with a 4-ft packed column of 2000 plates. In Chromatogram 5 the 51 peak maxima between *iso*pentane and *n*-nonane probably account for all the hydrocarbons present in proportions greater than about 0·3 vol per cent of the gasoline fraction, which constitutes about 20 vol per cent of the total crude oil.

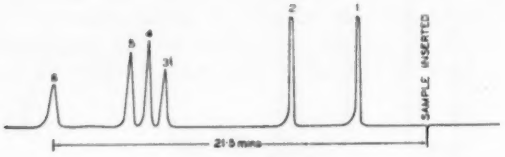
Although the high efficiency available with capillary columns reduces the importance of specificity in the stationary phase, this should not be over-emphasized.



CHROMATOGRAM 5

Straight-Run Benzine 45°–150° C Cut

Column:	120 ft 6 thou id glass
Stationary phase:	Squalane
Temperature:	70° C
Carrier gas:	Nitrogen
Inlet pressure:	10 psi
Flow rise:	0·25 ml/min
Sample size:	Approx 0·03 μg
Efficiency:	60,000 theoretical plates (*n*-butane)
Detector:	Flame ionization



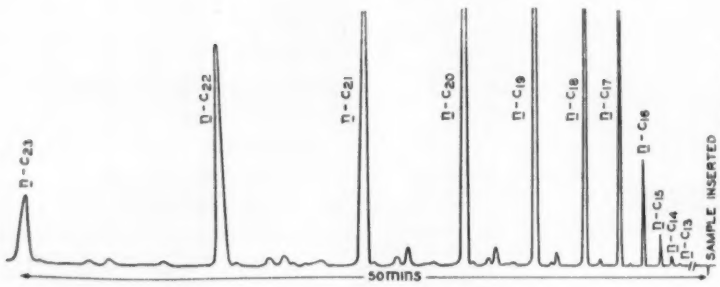
Components

1. Benzene
2. Toluene
3. Ethylbenzene
4. *p*-Xylene
5. *m*-Xylene
6. *o*-Xylene

CHROMATOGRAM 6

Separation of Aromatic Hydrocarbons

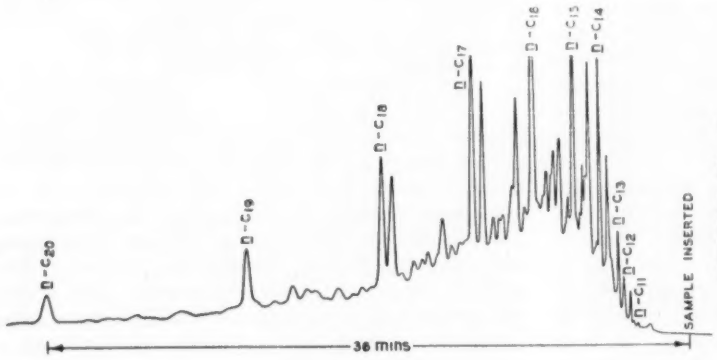
Column:	50 ft 10 thou id copper
Stationary phase:	7,8-Benzoquinoline (12 mg)
Temperature:	78·5° C
Carrier gas:	Nitrogen
Inlet pressure:	3 psi
Flow rate:	0·7 ml/min
Sample size:	Approx 1 μg
Efficiency:	14,600 theoretical plates (*o*-Xylene)
Detector:	Flame ionization



CHROMATOGRAM 7

Separation of a Urea Adduct from a Middle East Gas Oil

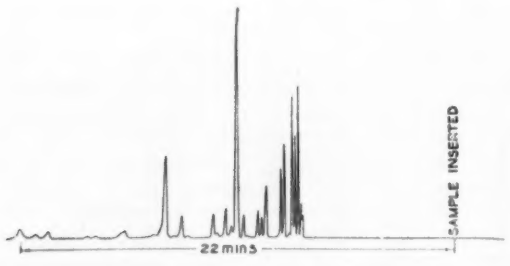
Column:	50 ft 10 thou id stainless steel
Stationary phase:	Apiezon grease L
Temperature:	240° C
Carrier gas:	Nitrogen
Inlet pressure:	1·7 psi
Flow rate:	0·50 ml/min
Sample size:	Approx 1 μg
Efficiency:	32,000 theoretical plates (n-C_{24})
Detector:	Flame ionization



CHROMATOGRAM 8

Crude Oil (Seepage Sample)

Column:	50 ft 10 thou id stainless steel
Stationary phase:	Apiezon grease L
Temperature:	205° C
Carrier gas:	Nitrogen
Inlet pressure:	2 psi
Flow rate:	0·50 ml/min
Sample size:	Approx 10 μg
Detector:	Flame ionization



CHROMATOGRAM 9

Separation of Volatile Components in Well Core Sample

Column:	160 ft 10 thou id stainless steel
Stationary phase:	Squalane
Temperature:	115° C
Carrier gas:	Nitrogen
Inlet pressure:	8 psi
Sample:	46 mg of crushed ore from well in West Africa
Preheating cycle:	200° C for 5 min
Detector:	Flame ionization

An example of this is in the analysis of mixtures of C_6–C_8 aromatic hydrocarbons. Separation of the xylene isomers with a non-polar stationary phase such as squalane requires upwards of 60,000 theoretical plates. As shown in Chromatogram 6, however, using 7,8-benzoquinoline as stationary phase the isomers are completely separated on a column having 14,600 plates in about 20 minutes. In an earlier publication [15] the authors have, in fact, shown that 7000 plates should be adequate.

At high temperatures a marked improvement over packed columns in the resolution of the high-boiling distillates is very obvious even with only a 50-ft column (*cf* [16]). In Chromatogram 7 of a carefully prepared urea adduct from gas oil, the separation between adjacent *n*-paraffins is several times the peak width, and a partially resolved pattern of six or seven weakly adducted hydrocarbons is seen at each carbon number.

The very small samples required, together with the high efficiency and rapidity of analysis, makes the use of capillary columns attractive in geochemical methods of oil prospecting. The authors have carried out some preliminary work on the examination of seepage samples and oil-bearing rocks. Chromatogram 8 shows the record obtained from a minute seepage sample too small even for a special micro-distillation technique used for such materials. It was, however, possible to determine an approximate carbon number distribution and a distillation curve from the chromatogram. The well-resolved pattern of peaks on the background hump of unresolved material is a little surprising with such a complex mixture. We have also investigated the possibility of obtaining chromatograms of the volatile components of oil-bearing rocks by preheating a small amount (50–250 mg) of the crushed rock to temperatures around 200° C and passing a pellet of nitrogen through it into the apparatus, via the normal sample introduction system. Chromatogram 9 shows the result obtained with about 50 mg of a crushed core from a well drilled into a potential source rock in Africa. The components eluted have not yet been identified, but it seems probable that they are mainly hydrocarbons.

DISCUSSION

Theoretical

Following the development of the theory of gas chromatography by Glueckauf [17] and van Deemter *et al*,[18,19] much effort has been expended in an endeavour to obtain high column efficiencies with packed columns. This work has been based on the van Deemter equation relating the height equivalent to a theoretical plate (HETP or H) to the linear gas velocity u. The most recent form of the equation [20] is as follows:

$$H = A + \frac{B}{u} + C_1 u + C_2 u \quad . \quad . \quad . \quad (1)$$

where $A = 2\lambda d_p$ (" Eddy " diffusion)

$B = 2\gamma D_g$ (longitudinal gaseous diffusion)

$$\left.\begin{aligned} C_1 &= 0{\cdot}01\left(\frac{k'}{1+k'}\right)^2 \frac{d_p^2}{D_g} \\ C_2 &= \tfrac{2}{3}\frac{k'}{(1+k')^2}\frac{d_f^2}{D_l} \end{aligned}\right\} \text{(non-equilibrium effects)}$$

A list of symbols is given at the beginning of the paper. The A term has been reduced by using supports of uniform particle size,[21–23] the B term by employing high average gas pressure,[21,23] and the C_2 term (due to resistance to mass transfer in the liquid phase) by reducing the proportion of liquid stationary phase [21,23] and by use of liquids having a poor solubility for the solutes.[23] The C_1 term (due to resistance to mass transfer in the gas phase) normally makes only a very small contribution to the total HETP. However, since it is directly proportional to the linear gas velocity, it determines, together with the B term, which is inversely proportional to u, the minimum HETP obtainable ($H°_{\min}$). This will be of the order of the particle diameter, as this approximates to the distance the gas must diffuse.[24] The lowest HETP value obtained with packed columns [21,23] seems to be about 0·4 mm with a particle diameter of approximately 0·1 mm.

The theory of coated capillary columns developed by Golay [2,3,25] results in an equation for the HETP in terms of u, for round tubes, of a similar form to that obtained by van Deemter:

$$H = \frac{B'}{u} + C'_1 u + C'_2 u \quad . \quad . \quad . \quad . \quad . \quad (2)$$

where $B' = 2D_g$ (longitudinal gaseous diffusion)

$$\left.\begin{aligned} C'_1 &= \frac{1 + 6k' + 11(k')^2}{24(1+k')^2} \cdot \frac{r^2}{D_g} \\ C'_2 &= \frac{(k')^3}{6(1+k')^2} \cdot \frac{r^2}{k^2 D_l} \end{aligned}\right\} \text{(non-equilibrium effects)}$$

The A term is, of course, zero, as there is only one gas path. The B' term is identical to van Deemter's B term, as the tortuosity factor γ is not applicable. In agreement with Golay we find C'_1 to be small but do not agree that there is a significant difference between packed and capillary columns in respect of this term. The width of the air peak with the former may well be confused by the use of detectors with moderately large dead volumes. The more important C'_2 term may be shown to be identical with van Deemter's C term as follows:

The ratio of the fixed phase capacity to the moving phase capacity (k') is related to the liquid and gas

cross-section (F_l and F_g) respectively by the equation

$$k' = \frac{F_l}{F_g} k. \quad . \quad . \quad . \quad . \quad . \quad (3)$$

For round capillary columns of radius r coated with a film of liquid d_f thick

$$\frac{F_l}{F_g} = \frac{\pi r^2 - \pi(r - d_f)^2}{\pi(r - d_f)^2} = \frac{d_f(2r - d_f)}{(r - d_f)^2}$$

but in general $r \gg df$

therefore

$$\frac{F_l}{F_g} = \frac{2d_f}{r}. \quad . \quad . \quad . \quad . \quad . \quad (4)$$

Substituting in equation (3)

$$k' = \frac{2d_f}{r} \cdot k \quad . \quad . \quad . \quad . \quad . \quad (5)$$

and

$$k = \frac{r}{2d_f} \cdot k' \quad . \quad . \quad . \quad . \quad (5b)$$

Substituting this value of k in the expression for C'_2 in equation (2) gives

$$C'_2 = \tfrac{2}{3} \cdot \frac{k'}{(1 + k')^2} \cdot \frac{d_f^2}{D_l}. \quad . \quad . \quad (6)$$

which is identical to the term obtained by van Deemter.

In Table III values of the coefficients for a typical packed column calculated from the van Deemter equation and values for a capillary column calculated from the Golay equation may be seen to be of similar order. It seems clear therefore that the same considerations apply to the performance of both packed and capillary columns. The minimum HETP ($H°_{min}$) with capillary columns is also determined by the combination of the B' and C'_1 terms, and using Golay's equation [2] the value of $H°_{min}$, assuming no mass transfer resistance in the liquid phase, may be shown to be

$$H°_{min} = 2\sqrt{B'C'_1} = 2\sqrt{\frac{2D_g r^2}{24D_g}} = 0{\cdot}58r \quad . \quad (7)$$

for the air peak ($k' = 0$).

The value of u at which this value of H is established is given by

$$u = \sqrt{\frac{B'}{C'_1}} = \sqrt{2D_g \cdot \frac{24D_g}{r^2}} = \frac{6{\cdot}9D_g}{r} \quad . \quad (8)$$

For components, which are well retained (k' large)

$$C'_1 \text{ tends to } \frac{11}{24} \cdot \frac{r^2}{D_g}$$

and

$$H°_{min} = 1{\cdot}9r \quad . \quad . \quad . \quad . \quad (9)$$

at

$$u = 2{\cdot}1\,\frac{D_g}{r} \quad . \quad . \quad . \quad (10)$$

TABLE III

Calculated Values for a Packed Column and a Capillary Column

	Packed column *	Capillary column
Stationary phase	Squalane	Squalane
Wt ratio solvent/support	20/100	—
Eluent gas	Nitrogen	Nitrogen
Solute	*n*-Butane	*n*-Butane
Partition coefficient at 20° C	40	40
F_l/F_g	$1{\cdot}5 \times 10^{-1}$	$3{\cdot}3 \times 10^{-2}$
k'	5·9	1·3
d_f (μ)	5	2
D_l (cm²/sec)	4×10^{-7}	4×10^{-7}
D_g (cm²/sec)	6×10^{-2}	6×10^{-2}
γ	0·7	1
λ	1·2	1
Particle size or capillary radius (μ)	440	120
A (cm)	$1{\cdot}1 \times 10^{-1}$	0
B (cm³/sec)	$8{\cdot}4 \times 10^{-3}$	$1{\cdot}2 \times 10^{-1}$
C_1 (sec)	$2{\cdot}3 \times 10^{-4}$	$5{\cdot}2 \times 10^{-4}$
C_2 (sec)	$5{\cdot}2 \times 10^{-3}$	$1{\cdot}6 \times 10^{-3}$

* Data taken from references 6, 11.

The $H°_{min}$ value for the air peak is the same as that given by Golay [2] and the column radius seems a useful criterion, as he suggests, for estimating $H°_{min}$ for components with a reasonable retention (*i.e.* k' from 0·5 to 15). With practical columns, where some degree of mass transfer resistance in the liquid phase is inevitable, the minimum HETP must be larger and occur at a lower linear gas velocity.

The HETP values given in Table II vary between four and fifteen times the column radius, and this may be attributed mainly to mass transfer resistance in the liquid film, but undoubtedly non-uniformity of the film and sample introduction difficulties are contributing factors. It is significant that the experimental value approaches nearest to $H°_{min}$ for the glass capillary, which has, as already mentioned, much the smoothest bore.

It is apparent therefore that a significant improvement in column efficiency can be obtained only by reducing the column radius, as this fixes the $H°_{min}$, which we have already approached to within a factor of four. For a particular column radius, however, it is desirable to reduce the resistance to mass transfer in the liquid phase by making the film as thin as compatible with an adequate plate capacity for sample detection. Golay's suggestion to increase the surface of the capillary seems very attractive, but is difficult to achieve in practice.

Having fixed the column radius and minimized the film thickness, the contribution of resistance to mass transfer in the gas phase to the HETP may be reduced by increasing the column average pressure, thus decreasing $D_g\left(D_g \alpha \frac{1}{p}\right)$. Since the minimum attainable HETP may be obtained only with values of u and D_g which satisfy the ratio

$$\frac{u}{D_g} = \sqrt{\frac{48r^2(1 + k')^2}{1 + 6k' + 11(k')^2}}$$

this reduction in D_g must be accompanied by a

corresponding reduction in u. This also leads to a smaller contribution by the C_2 term.

The effect of increasing the average column pressure is illustrated in Fig 6. Two HETP *v.* u curves have been constructed for a typical column run at average column pressures of 1 and 10 atmospheres. The reduction in HETP from $2{\cdot}7r$ to $1{\cdot}5r$, less than a factor of two, is only a small reward for the large increase in pressure which would involve considerable experimental difficulties. In addition, the minimum at the higher pressures is sharper, and since its position will change for different values of k' only a narrow range of components will be separated at maximum

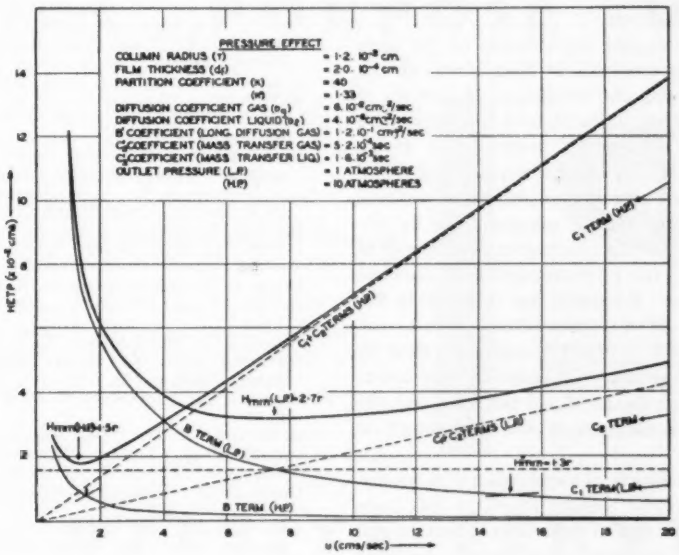

PRESSURE EFFECT
COLUMN RADIUS (r) = 1·2. 10⁻³ cm.
FILM THICKNESS (df) = 2·0. 10⁻⁴ cm
PARTITION COEFFICIENT (k) = 40
(k') = 1·33
DIFFUSION COEFFICIENT GAS (Dg) = 6. 10⁻² cm²/sec
DIFFUSION COEFFICIENT LIQUID (Dl) = 4. 10⁻⁶ cm²/sec
B COEFFICIENT (LONG. DIFFUSION GAS) = 1·2. 10⁻¹ cm²/sec
Cg COEFFICIENT (MASS TRANSFER GAS) = 5·2. 10⁻³ sec
Cl COEFFICIENT (MASS TRANSFER LIQ) = 1·6. 10⁻³ sec
OUTLET PRESSURE (L.P) = 1 ATMOSPHERE
(H.P) = 10 ATMOSPHERES
HETP (in 10⁻³ cms)
u (cms/sec)
C1 TERM (H.P)
C1+Cg TERMS (H.P)
Hmin(L.P)=2·7r
B TERM (L.P)
Cg+Cl TERMS (L.P)
Cg TERM
Hmin=1·3r
C1 TERM (L.P)
B TERM (H.P)

FIG 6

PRESSURE EFFECT

efficiency. Further, the reduction in the optimum average linear gas velocity from 7·5 to 1·3 cm/sec has the serious disadvantage that the time of analysis is increased by a factor of nearly six.

Summarizing: the low resistance to flow in capillary columns enables higher column efficiencies to be most easily achieved by increasing the column length. However, reduction in the HETP will be best made by:

1. Reduction of the column diameter.
2. Decreasing the film thickness.

Increase in the column average pressure accompanied by a reduction in linear gas velocity will only be worthwhile with very long columns having larger operating pressure drops.

Future Developments

Apart from detailed studies of the parameters affecting column performance along the lines discussed above, which will undoubtedly lead to a substantial improvement in column separation efficiency using liquid stationary phases, there remains the possibility of exploiting adsorption effects on the column wall, particularly for operation at high temperatures where liquids of low enough vapour pressure will be difficult to find. In this connexion the " structureless " surfaces described by White and Cowan [26, 27] are interesting.

The more defined geometry of capillary columns will allow absolute retention volumes and derived thermodynamic data on solution forces to be determined more accurately providing adsorption effects can be minimized. In addition, the fact that the film thickness can be measured with some accuracy will enable accurate values for the liquid diffusion coefficient to be obtained, as suggested by Keulemans.[24]

Much remains to be done, however, before capillary column gas chromatography becomes a routine analytical tool. It will be necessary to determine accurate absolute and relative retention volume data and to show that this may be related to similar data obtained on packed columns. In addition, more information on the response of the ionization detectors suitable for use with capillaries is required from both the point of view of linearity and sensitivity to molecules of different structural type, if much calibration work is to be avoided. The introduction of truly representative samples with mixtures having a wide range of volatility may well be difficult, and an isokinetic vapour division may be essential.

Potential Applications

The petroleum industry has been very active in the development of gas chromatography employing packed columns, and the technique is now applied over a wide area extending from routine gas analysis to the examination of paraffin waxes.

The rapidity of analysis and high column efficiency offered by capillaries should allow considerable extension of the application of the technique. The former will be of particular value in process monitor and control equipment and automatic laboratory analytical instruments, if high-speed automatic presentation devices based on integration of the detector output current with low loss polystyrene capacitors can be developed.

As mentioned earlier, the significance of the high column efficiencies is twofold, in that, first, at the lower molecular weights the resolution of isomers of similar structure can be achieved, and secondly, with the higher boiling fractions of petroleum a really powerful method of size separation is provided.

The authors' work with straight-run gasolines indicates that individual peak maxima can be obtained, with columns having efficiencies of the order of 100,000 plates, for all the predominant hydrocarbons present. For instance, Rossini [28] has isolated, in the 30°–150° C fraction of a representative crude oil, 84 hydrocarbons which probably constitute over 90 vol per cent of the fraction. Of these 84 only about 50 are present to more than 0·3 vol per cent and the total number of peak maxima in our chromatogram is of this order.

Work on the constitution of lubricating oils, waxes, and other high molecular weight fractions has been hampered by the difficulty of achieving a precise size separation of fractions containing compounds of similar molecular structure obtained by liquid chromatography, solvent extraction, extractive crystallization, thermal diffusion, etc. Packed column gas chromatography has already provided a significant improvement over molecular distillation, and capillaries, after some development, potentially offer another order of magnitude in separation efficiency. The very small quantities separated are, of course, a significant disadvantage, but the combination of mass spectrometry with capillary columns has exciting possibilities in this field.

CONCLUSIONS

Capillary column gas chromatography has great promise for the analysis of petroleum fractions and products, although considerable development is necessary before the technique reaches the status of a routine tool. In addition to straightforward analytical work this type of column has considerable possibilities as a research tool in investigating solution forces, adsorption effects, and the constitution of the very complex higher molecular weight fractions from petroleum.

ACKNOWLEDGMENT

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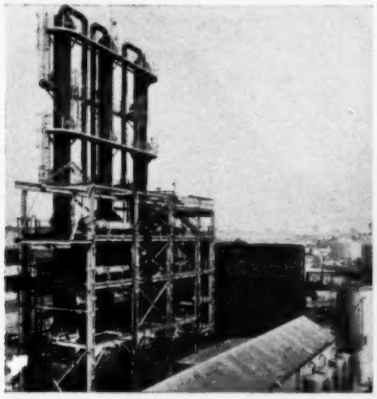

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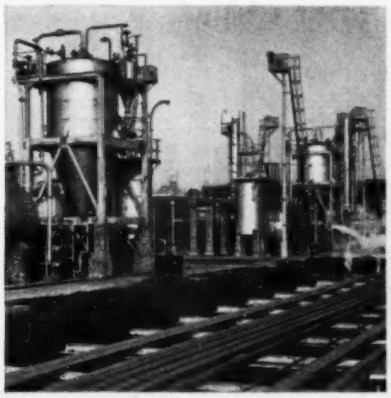

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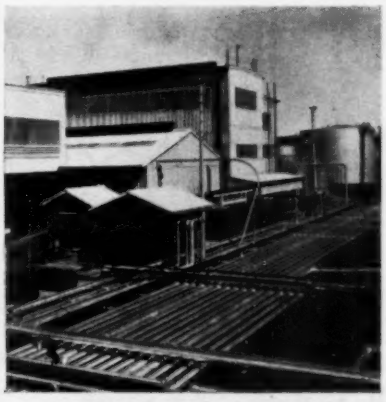

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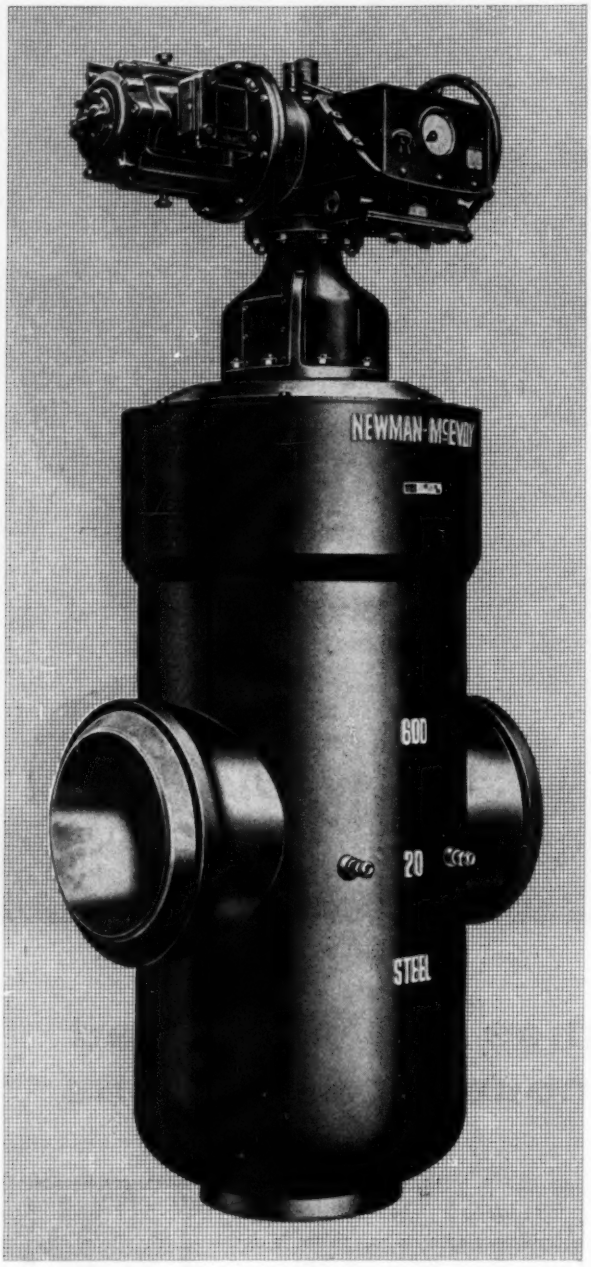

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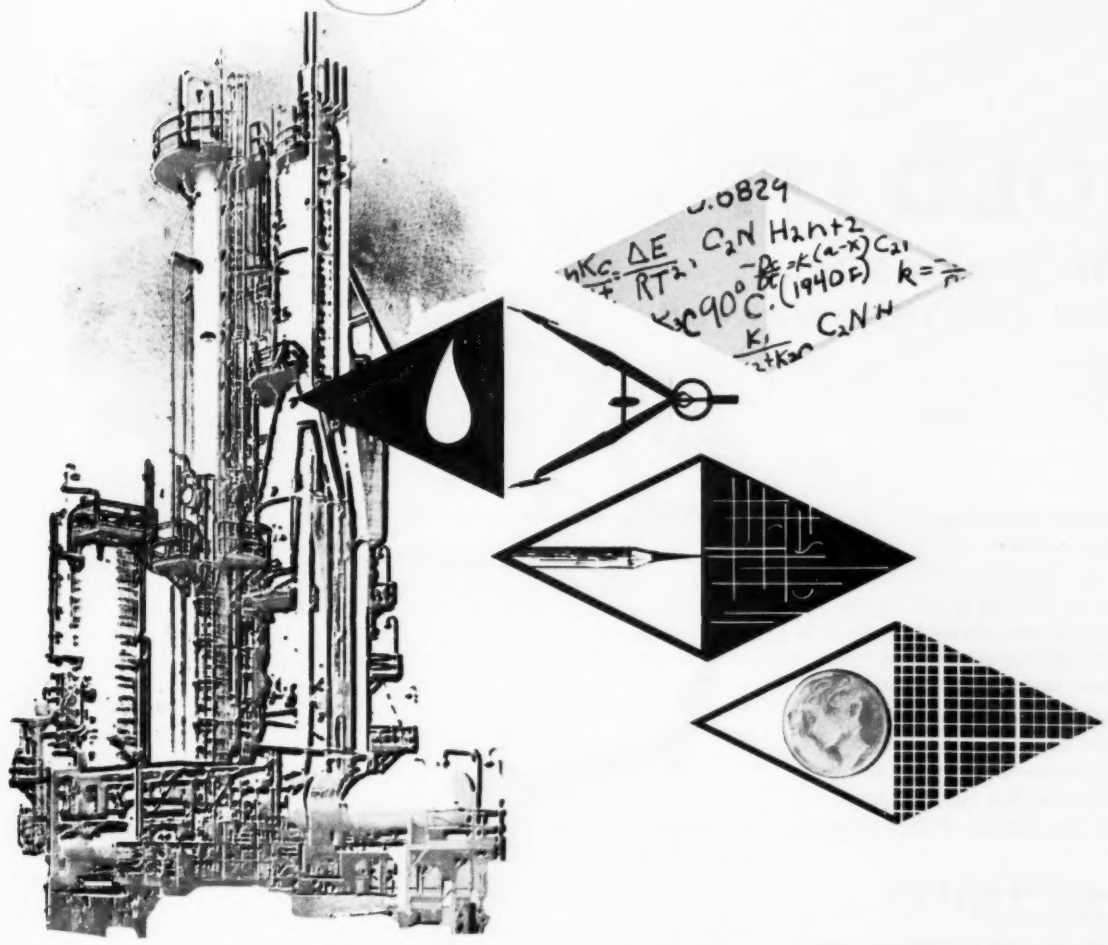

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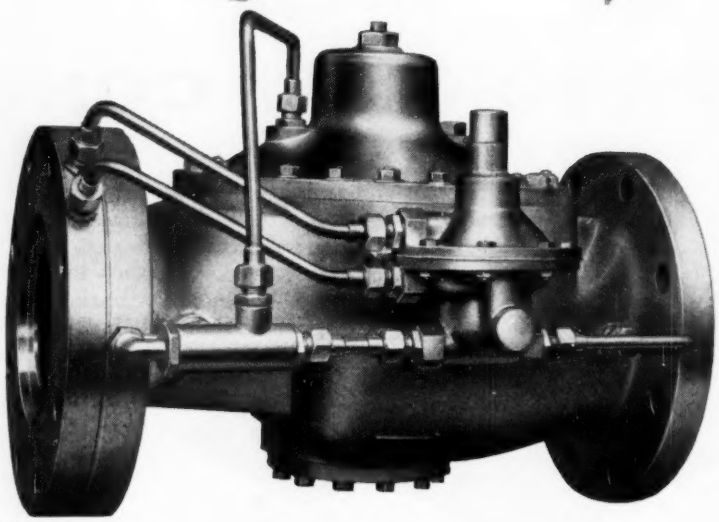

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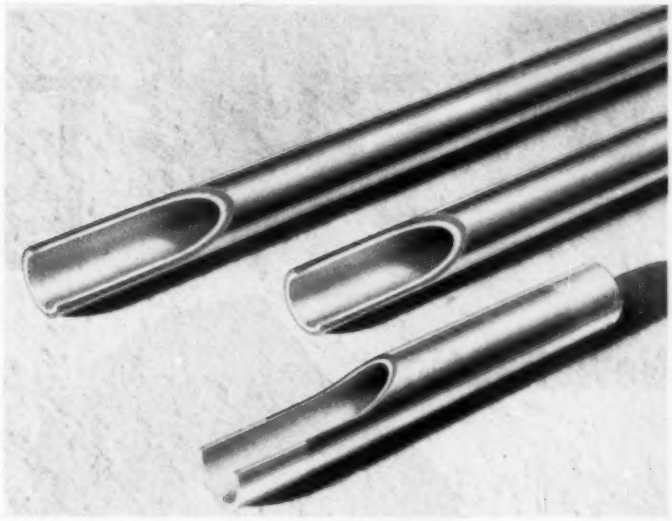

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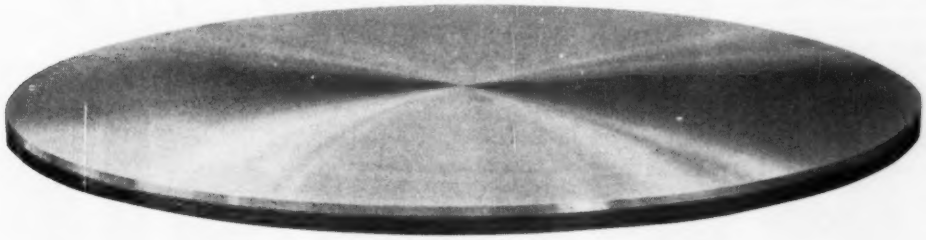

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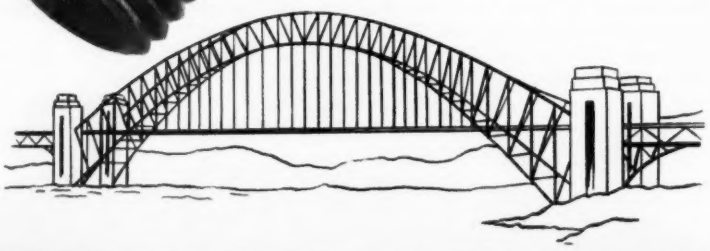

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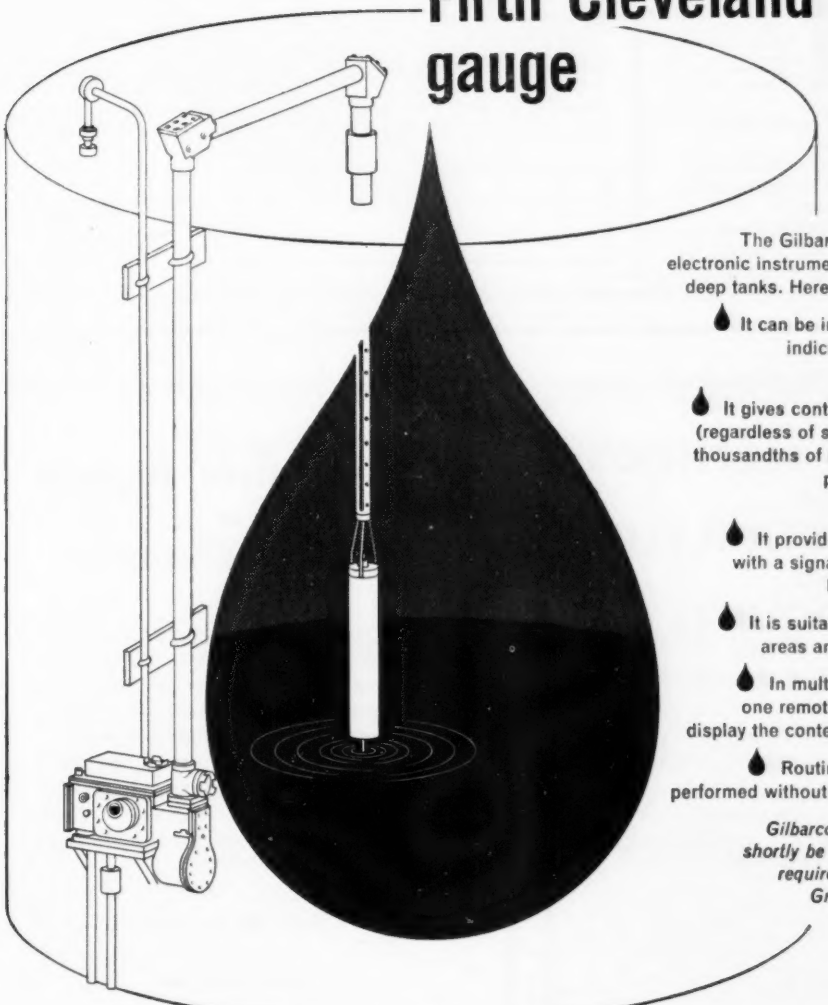

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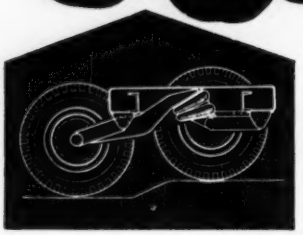

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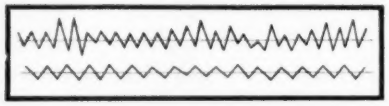

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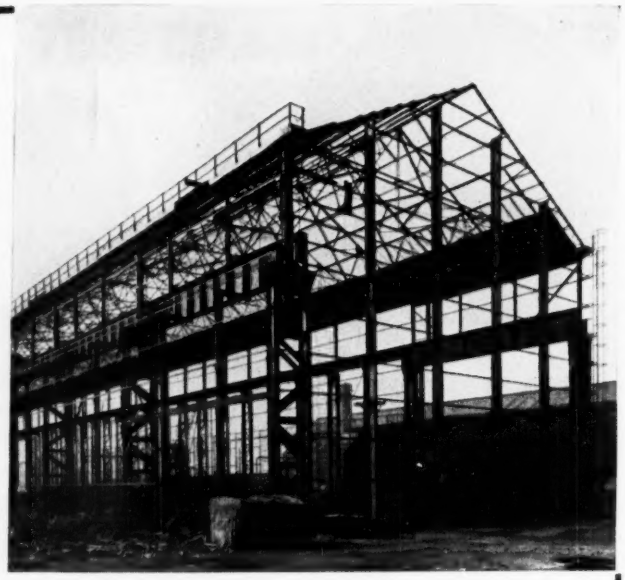

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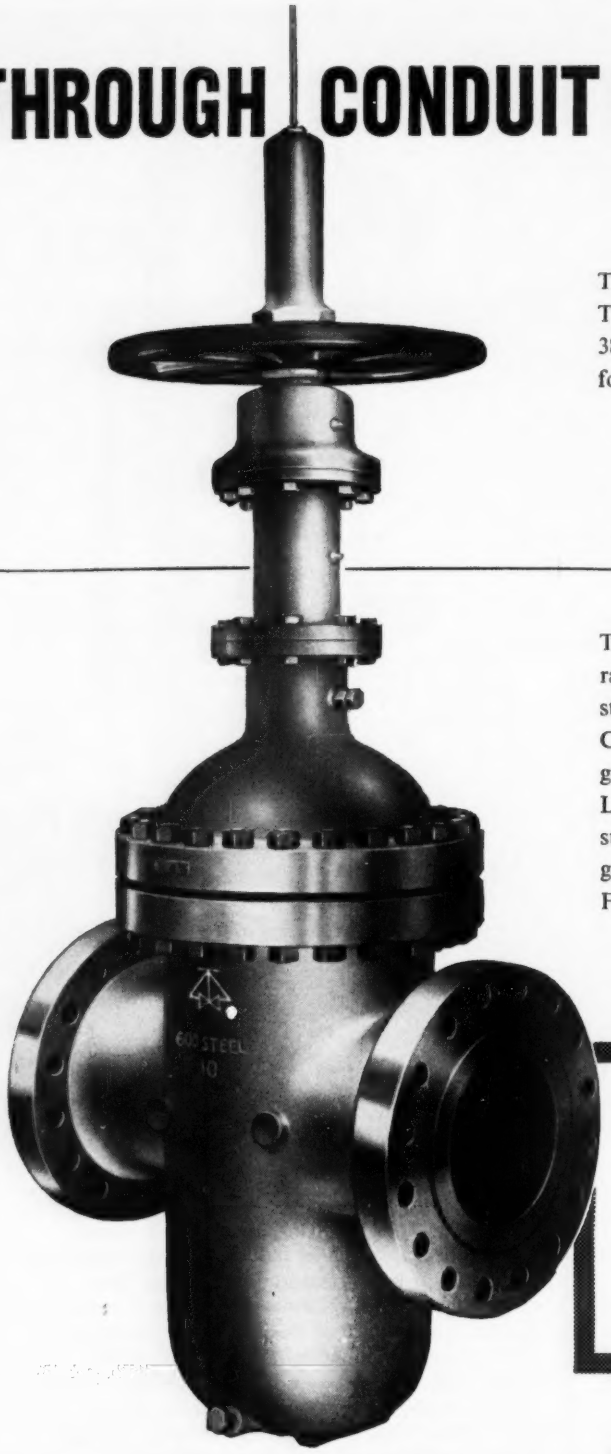

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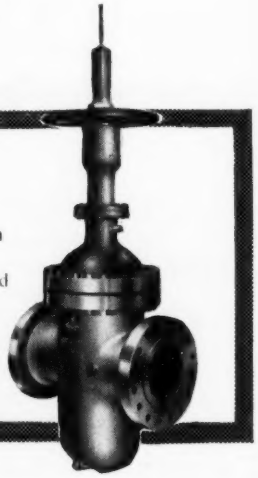

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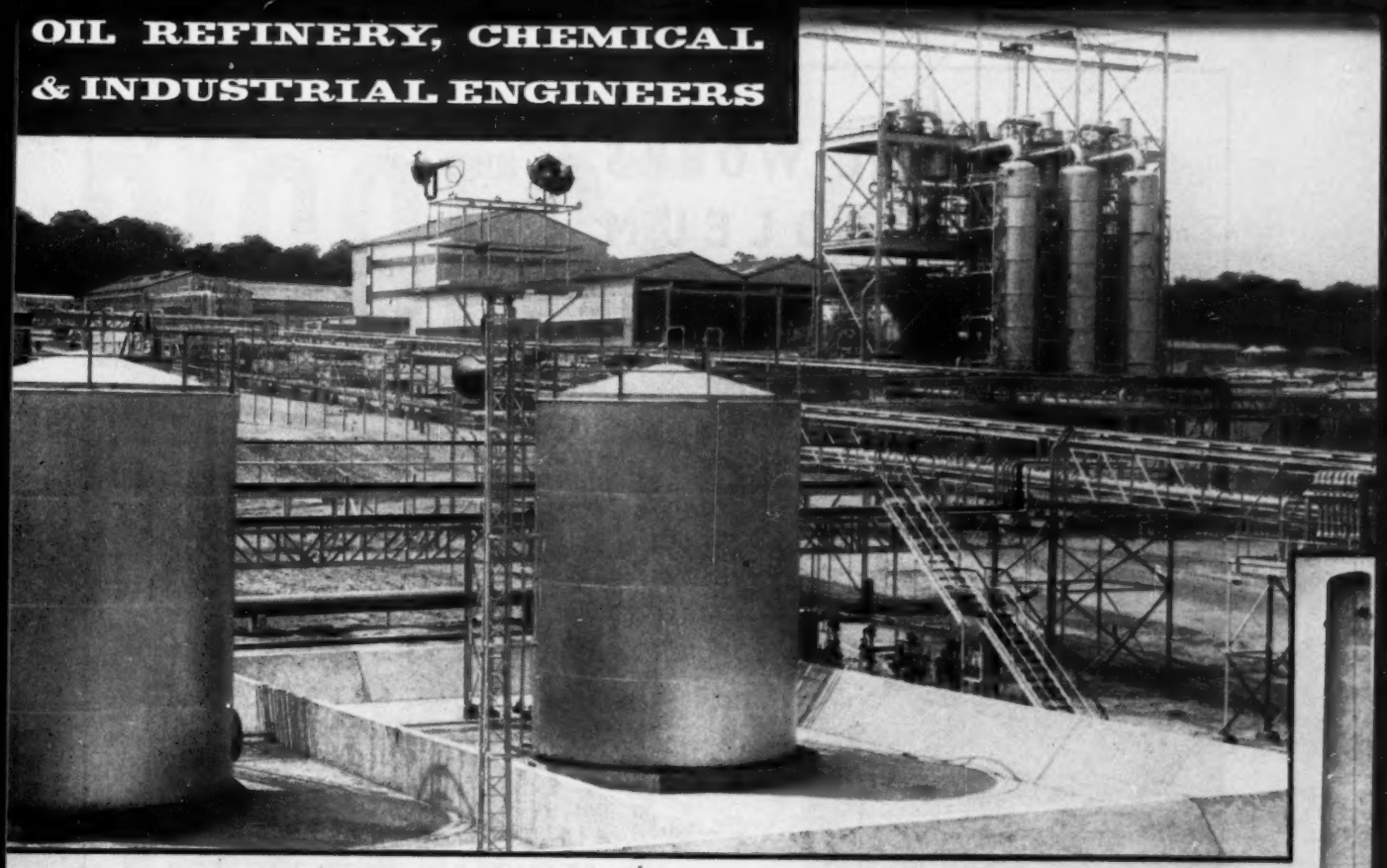

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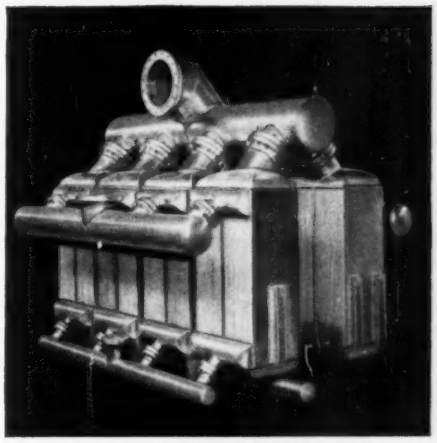

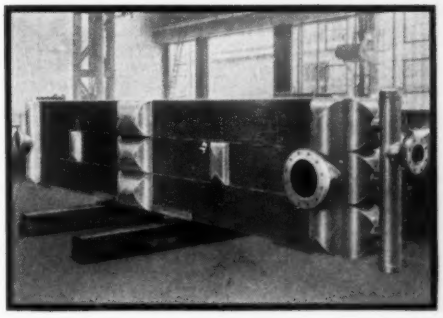

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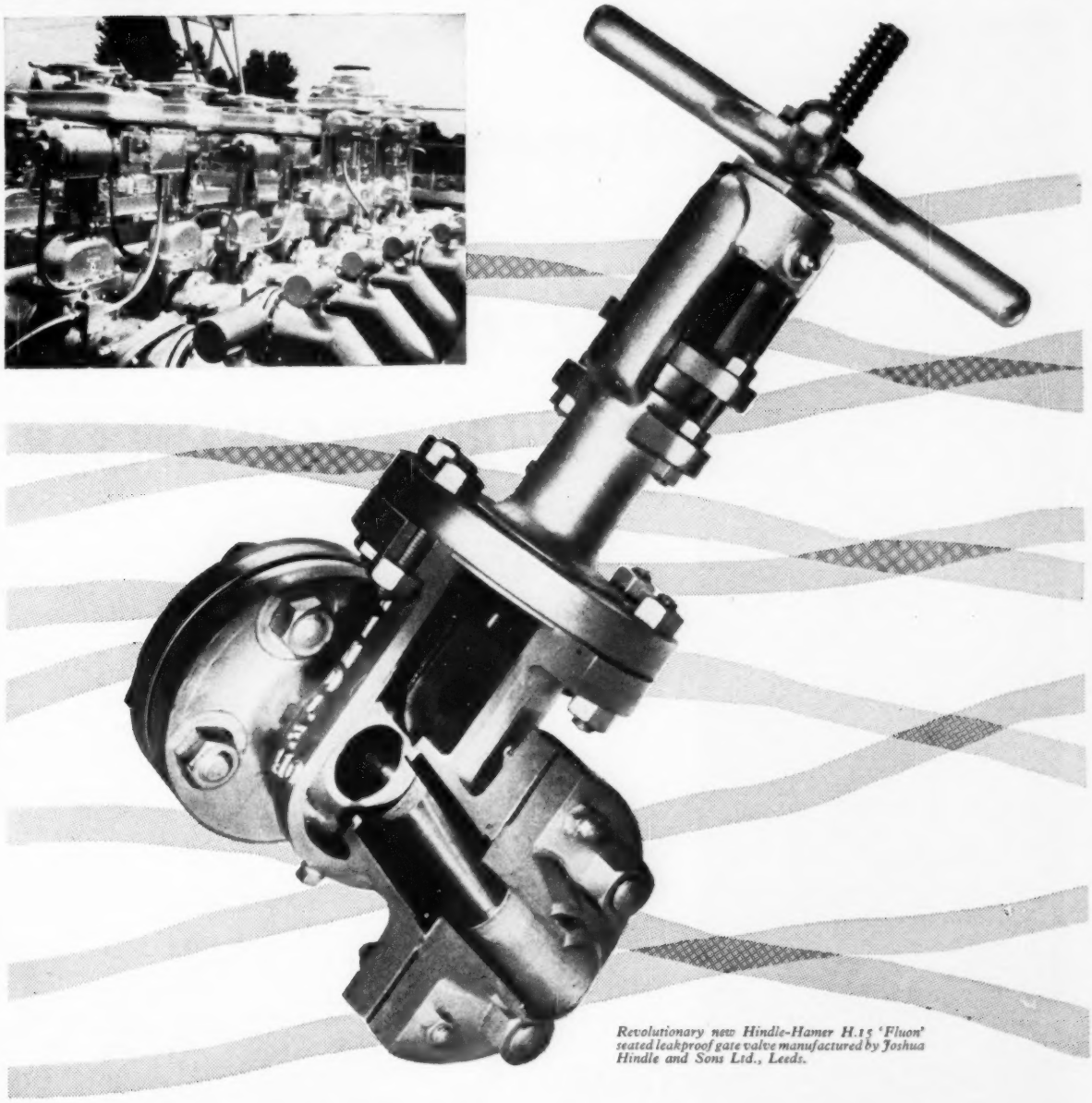

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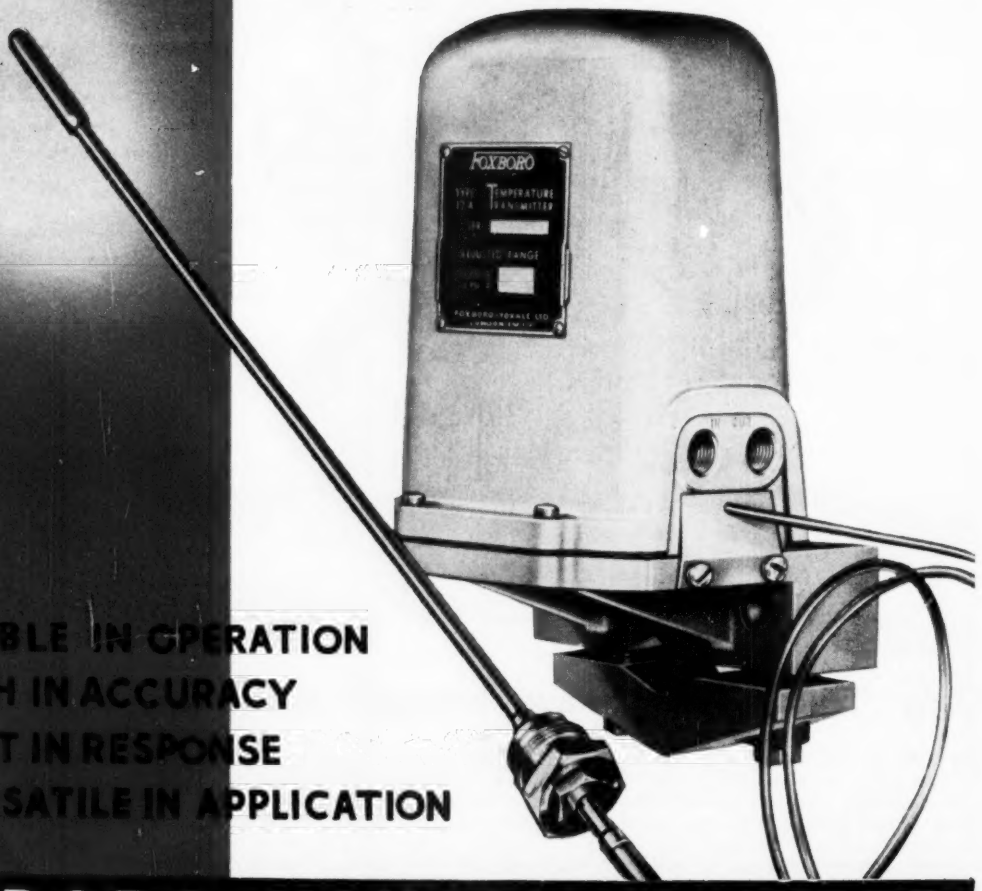

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